PB-228 718

DETERMINATION AND REMOVAL OF FLOATABLE MATERIAL FROM WASTE WATER

Engineering Science, Incorporated
Oakland, California

November 1965

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18 November 1965

Mr. Harry A. Faber, Chief
Research and Training Grants Branch
Division of Water Supply and Pollution Control
U.S. Public Health Service
Washington 25, D.C.

Dear Mr. Faber:

This will transmit our report for the contract years 1963-64 and 1964-65 pursuant to Contract WPD 12-01 (R_1)-63 and WPD 12-02-64, Division of Water Supply and Pollution Control, U.S. Public Health Service.

Analytical methods for the determination of floatable material and hexane extractable material have been developed. These methods have subsequently been used in bench scale evaluation of several schemes aimed at obtaining nearly complete removal of floatable material from wastes before their discharge into the marine environment.

Of the many treatment schemes tested, the concepts of pressure/vacuum flotation and of ordinary gravity flotation in combination with improved methods for the skimming of floatable material were shown to be the most promising. These findings are supported by surveys of actual treatment facilities aided by the developed analytical methods.

Finally, a sampling device has been developed which permits quantitative evaluation of the amount of floatable material in the ocean in the vicinity of existing outfalls.

The analytical methods are now ready to be used, and they will permit, for the first time, a complete evaluation on a quantitative basis of the problem of floatable material in marine disposal situations.

Sincerely yours,

Harvey F. Ludwig

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SUMMARY REPORT

Project Year 1963-1965

DETERMINATION AND REMOVAL OF FLOATABLE MATERIAL FROM WASTE WATER

Contract WPD 12-01(R_1)-63
Contract WPD 12-02-64

Prepared for

DIVISION OF WATER SUPPLY AND POLLUTION CONTROL

U.S. PUBLIC HEALTH SERVICE

By

ENGINEERING-SCIENCE, INC.

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November 1965

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DETERMINATION AND REMOVAL OF FLOATABLE MATERIAL FROM WASTE WATER

I. INTRODUCTION

THE MARINE DISPOSAL PROBLEM

The disposal of wastes into nearshore ocean waters is increasing in importance all over the world. One of the most important criteria for evaluating a system disposing domestic or industrial wastes into nearshore waters is the amount of floatable material in the waste.

Despite the importance of these floatable materials, most coastal treatment plants follow conventional designs, developed for inland disposal situations and placing emphasis on the removal of organic material and BOD without any special consideration to the problem of removal of floatable material. The objective of this study has been to develop treatment methods having this emphasis, thus producing an effluent suitable for discharge to open (unconfined) marine waters, and to develop quantitative methods for measuring floatables in wastes and in the marine receiving waters so that treatment plant performance may be properly evaluated.

The report describes in detail the progress made during the first two years of a planned three-year program aimed at the development of treatment methods which are to be oriented toward the removal of floatable material from domestic and industrial wastes before disposal of such wastes into the marine environment. It has been a special consideration during this work to develop treatment methods which will remove only the floatable material from a waste, followed by disposal of the remainder of the waste directly through a well designed and adequate outfall and diffuser system. Nevertheless, there are some situations (discharge to confined waters) where a more complete treatment of a waste may be warranted, especially with respect to settleable solids which might otherwise form objectionable bottom deposits, and combined removal of settleable solids and floatable material has therefore also been given due consideration.

ACKNOWLEDGEMENTS

The research staff wishes to thank the many people who have contributed in various ways to the project. Their advice and technical assistance have contributed very materially to the progress made. Special thanks are due to:

Dr. Erman A. Pearson	Professor of Civil Engineering University of California, Berkeley, and
Mr. Frank R. Bowerman	Assistant Chief Engineer, Los Angeles County Sanitation Districts

who have served as Special Consultants on the project.

Mr. John D. Parkhurst	Chief Engineer and General Manager, Los Angeles County Sanitation Districts, and
Mr. Norman B. Hume	Director, Bureau of Sanitation, City of Los Angeles

who have made the cooperation between their respective agencies and the project staff possible.

Mr. Raymond Stewart	Director of Laboratories, Los Angeles County Sanitation Districts,
Mr. Joe Nagano	Chief Chemist, Sewage Treatment Division, City of Los Angeles, and
Mr. Gerson Channin	Chief Sanitary Chemist, East Bay Mun. Util. Dist., Oakland, California

who have provided valuable assistance in appraising laboratory methods.

Mr. E. L. MacDonald, Jr.	Superintendent, Richmond Water Pollution Control Plant, Richmond, California, and
Mr. Henry Schweining	Superintendent, South San Francisco-San Bruno Water Pollution Control Plant

for their cooperation in the conduct of the treatment plant evaluations.

Mr. Walter E. Garrison	Deputy Assistant Chief Engineer, Los Angeles County Sanitation Districts, and
Mr. Wayne Messick	Field Engineer, Los Angeles County Sanitation Districts,

who have aided in the field evaluation of the ocean floatables sampler.

PROJECT STAFF

Representing Engineering-Science, Inc. on the project were Dr. Harvey F. Ludwig, Project Director; Mr. Ralf Carter and Dr. William Bishop, Project Supervisors; Mr. Jan Scherfig, Project Engineer responsible for the development of the analytical methods; Dr. R. Sage Murphy and Mr. Ye-shih Lin, Project Engineers responsible for the laboratory evaluation of the treatment methods; and Dr. Wonsuk Kim who, in cooperation with Mr. Ralf Carter, developed the ocean sampler. Mr. Mario Castillo provided valuable suggestions.

II. GENERAL CONSIDERATIONS, INVESTIGATION PLAN, AND STATEMENT OF PROGRESS

The objective of this study is to develop improved methods for the removal of floatable material from waste waters and to develop and collect background information which will permit a rational design of submarine waste disposal systems with respect to floatable material.

During the early part of the investigation it became clear from literature studies and field inspection of existing treatment units that the problem of removal of floatable material from waste water should logically be divided into two distinct groups. The first group consists of industrial wastes containing liquid floatable materials, especially hydrocarbon materials. The second group consists of both domestic and industrial wastes and is characterized by the content of particulate floatable material.

Because of the wide range of properties of industrial wastes, many of which would have no general significance, it was decided to concentrate the investigation on the removal of particulate floatable material from wastes mainly of domestic origin.

The evaluation of a particular marine waste disposal problem from the point of view of floatable material should ideally be based upon the following five main items:

1. Determination of the quantity of floatable material in the raw waste.
2. Determination of efficiency of removal of the floatable material from the waste by various treatment processes.
3. Determination of permissible concentrations of floatable material on the ocean surface in the vicinity of an outfall (a function of the beneficial uses of a given receiving water).
4. Determination of permissible concentrations of floatable material on the beaches in the vicinity of ocean outfalls and the rate of disappearence of these floatable materials on the beach.
5. Determination of the amount of material which is settleable at the time of discharge but which may be converted to floatable material in the ocean. (For example: grease particles adsorbed to settleable sludge may be liberated by mechanical or bacteriological action in the marine environment.)

On this basis possible combinations of treatment processes and ocean outfall systems can be analyzed on a cost-benefit basis and the optimal solution to a given problem determined as follows:

The quantity of floatable material in the raw waste combined with known treatment efficiencies permit the determination of the concentrations and amounts of floatable material in the effluent for different treatment processes.

From this knowledge in combination with the usual information about the ocean current distribution in the receiving area, it is then possible to design a diffuser system which will permit the discharge of the waste without exceeding permissible concentrations of floatable material in a given area.

Again from knowledge of the ocean current distribution combined with a two-dimensional diffusion analysis analog to the three-dimensional diffusion analysis used in problems involving soluble wastes, the amount of floatable material reaching the shoreline can be determined as a function of the distance from the diffuser system to the shore.

It is necessary to determine the rate of disappearance of the floatable material from the beach. From the permissible concentration of floatable material on the beach (the steady state concentration) and the rate of disappearance of the floatable material, the maximum permissible amount of floatable material which may reach the beach per unit time can be calculated. This value combined with the results of the above mentioned two-dimensional diffusion analysis in turn determines the required length of the outfall for each set of combinations of treatment methods and diffuser systems.

INVESTIGATION PLAN

Of the above mentioned five main parts of the overall problem of disposal of wastes into the marine environment, the problem of quantitative determination of floatable material, the problem of efficiency of floatables removal methods, and the problem of quantitating the amounts of floatable material on the ocean surface were considered the most important. In addition, it was found that there was a need for a more convenient method of extracting oil and grease from liquid wastes than the existing wet and dry extraction methods.

Based upon the above considerations, the following three-year plan of investigation was decided upon:

1. Develop improved method for extraction of "oil and grease".
2. Develop method for quantitative determination of particulate floatable material.
3. Investigate on a laboratory and bench scale methods for removal of floatable material.
4. Determine in a sufficiently large pilot plant the removal efficiencies and process characteristics of the one or two most promising bench scale results.

5. Concurrent with 1. and 2. a floatables sampler should be developed which would permit quantitative evaluation of existing ocean disposal systems and at least lead to knowledge of the order of magnitude of the concentration of floatables permissible in the vicinity of an outfall, preferably on a mass per unit area basis.

STATEMENT OF PROGRESS

There have been no large deviations from the general plan of investigation outlined. During these first two years of research, a method for determination of hexane extractable material and for determination of particulate floatable material has been developed and put on a firm statistical basis.

Several methods for removal of floatable material including both biological and physical systems have been tested on a laboratory scale. The two most promising were found to be vacuum flotation (dissolved air flotation) and conventional gravity flotation in combination with improved skimming systems.

The laboratory results were supported by surveys of two operating treatment plants employing vacuum flotation and conventional flotation, respectively.

Several systems for sampling of floatable material in the ocean were investigated and a system finally developed which will enable quantitative evaluation of floatable material. Plans are now being made for conducting surveys of submarine disposal systems in cooperation with several California agencies. The purpose will be to collect the necessary background information permitting the determination of allowable concentrations of floatable material on the ocean surface.

Likewise, plans are presently being made for the final evaluation on a pilot plant scale of the two treatment methods selected on the basis of the bench scale data.

III. DETERMINATION OF HEXANE EXTRACTABLE MATERIAL IN WASTE WATERS

INTRODUCTION

The hexane extractable compounds present in domestic sewage consist primarily of fats, free fatty acids, and hydrocarbons of petroleum origin. Metal salts of the fatty acids are not soluble in hexane and must be converted to their corresponding free fatty acids before extraction.

Although several methods were available for the determination of Hexane Extractable Material (HEM) in raw and treated sewage, they all had one or more serious disadvantages, and an effort was therefore made to develop an improved HEM method.

REVIEW OF EXISTING METHODS

Two methods have been adapted for use as standard methods in the United States (Reference 1). The first is based upon concentrating dissolved, emulsified, and suspended oil and grease on a diatomaceous earth filter, followed by drying. The subsequent extraction of the filter aid and the suspended material can be performed in an extraction thimble or by mechanical stirring of the filter in hexane. The second method is mainly used for sludge and sediments, and consists in mixing an acidified sludge with magnesium sulfate, monohydrate, followed by extraction of the crushed mixture in an extraction thimble.

Preliminary investigation of these methods showed that although the reported precisions and accuracies could be obtained using standards of motor oils and fats, the methods were both time and labor consuming. Subsequent investigation for a more convenient method also revealed that the above procedures may give low recoveries of soaps due to inadequate acidification procedures.

EXPERIMENTAL WORK

The extraction method suggested by Pearson and Thomas (Reference 2) appeared to be the most promising. The method consisted of placing a 150 ml acidified sample in a flask below a condenser provided with a central recycling tube. The solvent was placed in a pre-weighed flask on a hot plate. The solvent evaporates from the flask, is condensed, and cycles through the sample before it overflows to the solvent flask.

A series of preliminary tests were run on standards containing fats (Crisco) and soaps, but unfortunately the recoveries were found to be very inconsistent, as low as 50 percent.

In order to determine the reason for these low and inconsistent recoveries, the procedure was divided into the following three major substeps:

Extraction
Solvent Evaporation and Residue Weighing
Acidification and Soap Conversion

and each step was investigated separately. The results were then combined into an overall procedure and its standard deviation determined on several types of raw and treated sewage.

Extraction:

Modification of Extraction Apparatus: One disadvantage of the extraction apparatus as proposed by Pearson (Reference 2) was the relatively small volume of sewage (150 ml) which could be extracted. The extraction apparatus was therefore redesigned and provided with a removable 1-liter extraction chamber in the form of a glass flask with a standard glass joint. The extraction apparatus is shown in Figure III-1 (photo of the unit) and in detail in Figure A-1 in Appendix A. The flasks are used directly as sampling containers, transported to the laboratory, and extracted without any need for time consuming and inaccurate transfer operations.

It was believed that one of the reasons for the low recoveries in the preliminary tests was inadequate contact between the solvent and the sample during the extraction. The unit was therefore provided with a magnetic stirrer using a Teflon-coated 1.5 inch long stirring bar.

Extraction of Fats and Soaps with Modified Extractors: A series of extraction tests were made on a combined standard emulsion containing fats (Crisco) and calcium stearate, using the modified extraction apparatus. The results from these tests showed that the modifications introduced increased the recovery to between 70 and 80 percent as compared to approximately 50 percent before, but without any increase in precision. The reason for the low recovery was not evident, but a possible explanation was that the soaps were not completely converted to the corresponding fatty acids by just adding 10 ml conc. hydrochloric acid per liter before extraction as described in Standard Methods (Reference 1). To prove this hypothesis, a series of extractions were made on standards containing from 6 mg/l to 100 mg/l of fat (Crisco) but no soaps. The results are reported in Table III-I and show an average recovery of 98.5 percent with a coefficient of variation of 0.024. In addition, two samples of free fatty acids were extracted with an average recovery of the free fatty acids of 98.7 percent.

These two sets of results showed that the modified extraction procedure is adequate for extraction of hexane soluble fats.

Solvent Evaporation and Residue Weighing

In order to minimize weighing errors, the hexane with the extracted material was transferred to an aluminum cup (Van Waters and Rogers Catalog 63 #25433) before final evaporation and weighing as proposed by Jenkins (Reference 3). However, it was found that one cup was not satisfactory due to occasional losses of residue by creeping of the extract during the evaporation process. The use of two cups, one inside the other, eliminated these losses.

FIGURE III-1

GREASE EXTRACTION APPARATUS

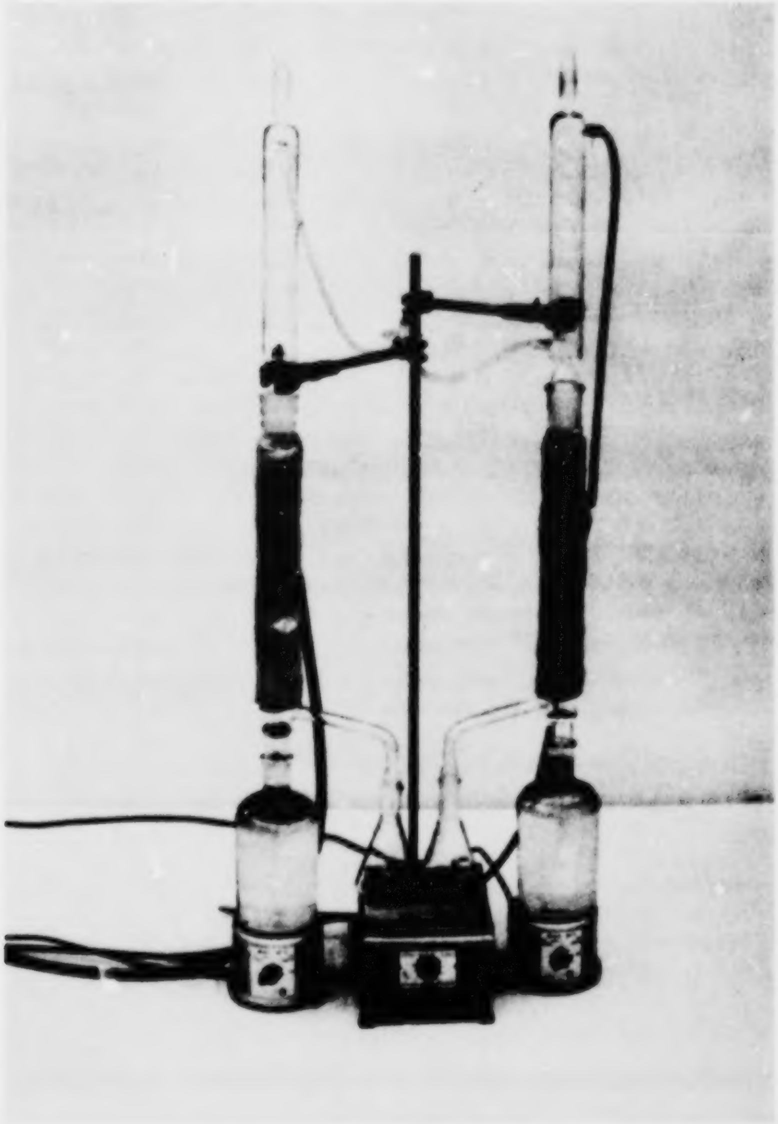


TABLE III-I

RECOVERY OF FATS BY LIQUID EXTRACTION OF ONE LITER SAMPLES

No.	Fats Added (g)	Fats Recovered (g)	Average Blank (g)	Net Recovery (g)	Recovery Percent
1	0.1038	0.1040	0.0010	0.1030	99.2
2	0.1038	0.1042	0.0010	0.1032	99.4
3	0.0811	0.0820	0.0010	0.0810	99.9
4	0.0762	0.0762	0.0010	0.0752	99.7
5	0.0759	0.0769	0.0010	0.0759	100.0
6	0.0542	0.0523	0.0010	0.0523	96.5
7	0.0542	0.0548	0.0010	0.0538	99.2
8	0.0152	0.0163	0.0010	0.0153	99.5
9	0.0152	0.0161	0.0010	0.0151	100.5
10	0.0162	0.0169	0.0010	0.0159	98.2
11	0.0062	0.0023	0.0010	0.0013	(21.)*
12	0.0062	0.0067	0.0010	0.0057	92.0

Average Recovery: 98.5% (*Sample No. 11 not included).

Coefficient of variation: 0.024.

Several modifications have been used and proposed for use in the evaporation of the hexane before weighing the residue, but no information was found about their efficiencies. Since the evaporation was considered to be an important step in the overall procedure, a series of tests were carried out as follows to determine the most accurate and fastest drying procedure.

Solutions of fat (Crisco) and of stearic acid were made up in hexane, each containing about 30 mg fat or stearic acid per 20 ml hexane. Twenty milliliters of the hexane solution were pipetted into preweighed aluminum cups and the hexane allowed to evaporate to dryness at room temperature either at a pressure of 760 mm Hg or 40 mm Hg. The evaporation period was then followed by a 0, 5, or 15 minute drying period at 85° C. In addition to these tests, six samples were placed directly in the 85° C oven for 40 minutes without previous evaporation at room temperature. The results are reported in Table III-II. The experiment showed no difference between evaporation at 760 mm Hg and at 40 mm Hg. Comparison of the effect of drying time in the 85° C oven shows that there is a significant difference between no drying and drying for 5, 15, and 40 minutes, respectively, but that there was no difference between 5, 15, and 40 minutes drying time. The following drying procedure was therefore decided upon (for details see Appendix A):

1. Concentration of hexane extract in the Erlenmeyer flask by boiling off hexane to 10 ml on a steam bath.
2. Quantitative transfer to preweighed aluminum cups.
3. Evaporating at room temperature in a hood.
4. Five minutes drying at 85° C.

Subsequent use of this procedure on sewage samples showed that reproducible results could be obtained. Additional drying at 85° C for 5 minutes results in weight changes of less than 0.3 mg for most sewage samples.

Acidification and Soap Conversion

Preliminary Investigation: The third step in the evaluation of the extraction procedure was the conversion of metalsoap to the corresponding free fatty acids. As pointed out by Ludwig (Reference 4), the methods generally used may not be adequate for the conversion of soaps to fatty acids. The following experiments were therefore conducted:

Twelve samples of 100 mg sodium stearate were dissolved in 100 ml distilled water and calcium stearate precipitated by addition of calcium chloride. Three ml of concentrated hydrochloric acid or sulfuric acid were added to each suspension. Following the acidification, four of the suspensions were heated to 100° C and kept at that temperature. The remaining eight were kept at room temperature. The suspensions were examined after 1, 3, 4, 5, and 36 hours, respectively, whereafter it was determined whether the calcium stearate had been converted to stearic acid. This was done by heating the suspensions rapidly to approximately 90° C and observing whether the suspended material would melt. (Stearic acid has a melting

TABLE III-II

EFFECT OF EVAPORATION PRESSURE AND DRYING TIME ON RECOVERY OF FATS AND FATTY ACID FROM HEXANE

Material Added	Weight Added mg	Evaporation Pressure (mm Hg)	Drying Time minutes at 85° C	Weight Recovered (mg)	Percent Recovery
Fat	30.2	Direct to oven	40	30.3	100.3
Fat	30.2	40	5	30.3	100.3
Fat	30.2	760	15	30.3	99.3
Fat	30.2	40	Not dried	30.9	102.3
Fat	30.2	Direct to oven	40	29.6	98.0
Fat	30.2	760	Not dried	32.5	107.6
Fat	30.2	760	5	30.5	101.0
Fat	30.2	Direct to oven	40	30.1	99.7
Fat	30.2	40	15	30.8	102.0
Fatty Acid	28.5	Direct to oven	40	28.0	101.0
Fatty Acid	28.5	40	5	28.9	101.4
Fatty Acid	28.5	760	15	28.5	100.1
Fatty Acid	28.5	40	Not dried	27.9	97.9
Fatty Acid	28.5	Direct to oven	40	29.0	101.8
Fatty Acid	28.5	760	Not dried	(79.5)	---
Fatty Acid	28.5	760	5	28.9	101.4
Fatty Acid	28.5	Direct to oven	40	28.4	99.6
Fatty Acid	28.5	40	15	29.0	101.8

point of 69° C compared to 179-180° C for calcium stearate.) The results are reported in Table III-III and show that there is a complete conversion of soaps to stearic acid within one hour when heated to 100° C in the presence of either H_2SO_4 or HCl, whereas acidification for up to five hours at 20° C does not result in complete conversion.

Quantitative Results: Based upon the preliminary results, a quantitative extraction experiment was performed to determine the minimum time and temperature required for complete conversion of soaps. It was believed that the temperature should be kept as low as possible to avoid significant side reactions, but also that the temperature should be above the melting point of stearic acid to insure continuous removal of converted material from the surface of the grease particles.

Known quantities (about 100 mg per liter) of calcium stearate were suspended in one liter of distilled water, acidified with 10 ml conc. HCl, and placed in a 72-75° C water bath for 0, 1, and 2 hours. After preheating, the samples were extracted for 1, 2, 3, or 4 hours, and the percent recovery determined (corrected for the hexane blank and converted to calcium stearate). The results are reported in Table III-IV and show a minimum of one hour preheating at 70° C followed by two hours extraction will result in almost complete recovery of soaps. Because subsequent observations showed that not all extractor units begin extraction simultaneously due to differences in heating, etc., a three-hour extraction period has been used throughout the study.

Overall Evaluation of Extraction Procedure

The results were combined to a complete procedure as described in detail in Appendix A. The basic steps include acidification with 10 ml conc. HCl or H_2SO_4 per liter, preheating at 72° C - 75° C for one hour, extraction with hexane for three hours and drying the residue for five minutes at 85° C.

A series of hexane extractions were made on different types of homogenized sewage, and the standard deviation of the method determined as follows: For each of the different types of sewage a 15-liter sample was obtained, homogenized in a Waring Blendor and placed in a five gallon jug provided with a stirrer. The number of 1-liter samples to be analyzed was withdrawn from the jug at the same time and extracted simultaneously. The results are given in Table III-V and show the standard deviation to be approximately 1 mg per liter of extractable material for sewage containing between 30 mg/l and 100 mg/l floatable material. No attempt was made to determine the standard deviation of non-homogenized sewage since this would mainly reflect the variation in a particular sewage, not the variation in the method.

Additional Investigations Related to the Hexane Extraction Procedure

Compositing Samples for Hexane Extractions: Although the method of compositing and stabilizing sewage samples for analysis of oil and grease by acidification has been used widely, a literature survey did not reveal any data showing its reliability.

TABLE III-III

CONVERSION OF SOAP TO FATTY ACID BY TREATMENT WITH CONCENTRATED ACID

About 100 mg Calcium Stearate suspended in 100 ml distilled water and acidified with 3 ml concentrated acid.

Temperature of Conversion °C	Time Elapsed Hours	Acid (3 ml)	Result of Conversion
20°	1	H_2SO_4	Not Converted
20°	1	HCl	Not Converted
20°	3	H_2SO_4	Not Converted
20°	3	HCl	Sample Lost
20°	5	H_2SO_4	Not Converted
20°	5	HCl	Not Converted
20°	36	H_2SO_4	Converted
20°	36	HCl	Not Converted
100°	1	H_2SO_4	Converted
100°	1	HCl	Converted
100°	4	H_2SO_4	Converted
100°	4	HCl	Converted

TABLE III-IV

EFFECT OF PREHEATING AND EXTRACTION TIME ON RECOVERY OF CALCIUM SOAP

Reported Values are Percent Recovery
(Concentrations approximately 100 mg per liter)

Extraction Time	Preheating Time at 72°-75° C 0 hrs	 1 hr	 2 hrs
1 hour		88.3%	83.5%
2 hours		96.3%	96.2%
3 hours		96.2%	95.5%
4 hours	74% & 64.2%		

TABLE III-V

THE STANDARD DEVIATION OF HEXANE EXTRACTION METHOD

(Determined on replicates of homogenized sewage)

Type of Sewage	Number of Replicates	Average conc. of Hexane Extractables mg/l	Standard Deviation mg/l	Coefficient of Variation (Percent)
Raw domestic	7	37.7	0.7	1.9
Raw combined domestic & industrial	5	102.7	0.9	0.9
Primary effluent from domestic	4	28.7	1.0	3.5

Since a compositing procedure was intended for use in a subsequent plant survey, it was decided to determine the adequacy of the acid compositing procedure.

A 15-liter sample of raw domestic sewage was collected and acidified by adding 10 ml conc. HCl per liter. Duplicate 1-liter samples were analyzed both immediately after collection, and after several days, using the procedure described in Appendix A. The results presented in Table III-VI show no significant change in the concentration of Hexane Extractable Material over a period of 168 hours. The determinations were made on non-homogenized samples which account for the larger variation than obtained with homogenized samples.

TABLE III-VI

CHANGE IN CONCENTRATION OF HEXANE EXTRACTABLE MATERIAL WITH TIME

Samples preserved with 10 ml conc. HCl per liter

Time	Hexane Extractable Material, mg/l	
0 hrs	47.1	Average 48.4 mg/l
	49.7	
72 hrs	46.2	
	45.5	Average 45.8 mg/l
120 hrs	52.1	
	47.6	Average 49.9 mg/l
	50.0	
168 hrs	47.9	
	47.6	Average 48.8 mg/l
	50.7	

Quantitative Effect of the Preheating Procedure

The effect of the preheating procedure on the amount of Hexane Extractable Material (HEM) was determined on samples of raw sewage.

The sewage samples were analyzed according to the procedure given in Appendix A, and the results were compared with the results obtained when the preheating step was omitted and the samples, instead, were extracted for four hours directly after acidification. Although statistically significant results were not obtained, the results in Table III-VII indicate that there is a positive relation between the amount of Hexane Extractable Material and preheating.

The results indicate that the effect of preheating is more pronounced with increasing amounts of Hexane Extractable Material. The additional amount of fatty acid extracted after preheating proved to be relatively small compared to the total amount of Hexane Extractable Material in at least these three types of sewage. No additional efforts were therefore invested in the investigation of the Hexane extraction procedure.

TABLE III-VII

EFFECT OF PREHEATING (1 HOUR AT 72° C) ON AMOUNT OF HEXANE EXTRACTABLE MATERIAL

Type of Sewage (All Raw)	1 hr Preheating 72° C 3 Hrs Extraction		No Preheating 4 Hrs Extraction	
Domestic	47.1 49.7	Avg. 48.4 1)	45.6 44.8	Avg. 45.2
Domestic	54.2 52.2	Avg. 53.2	53.1 54.0	Avg. 53.5
Domestic-Industrial	51.6 50.4	Avg. 51.0	42.6	
Domestic-Industrial	39.2 39.8	Avg. 39.5	38.9 37.9	Avg. 38.4
Domestic-Industrial	80.4 81.2	Avg. 80.8	75.8 74.6	Avg. 75.2
Mainly Industrial	5 Samples, Avg. 103. Standard Dev. 0.9		94.6 91.1 (83.8)	Avg. 92.8

1) Values in Table are mg Hexane Extractable Material per liter sewage. All determinations were made in duplicate. Both single and avg. values given.

Composition of the Hexane Extract

The purpose of this experiment was to determine whether the increase in Hexane Extractable Material obtained after preheating consisted in free fatty acids as indicated by the preliminary work, or whether it was due to side reactions producing compounds unrelated to "Oil and Grease".

A sample of sewage was extracted in three different ways:

a. No HCl added, 1 hr preheating, 3 hrs extraction

b. 10 ml HCl added, 1 hr preheating, 3 hrs extraction

c. 10 ml HCl added, no preheating, 4 hrs extraction

and the total amount of Hexane Extractable Material determined. The extracts were then redissolved quantitatively in carbon disulfide, and the concentrations of free fatty acids C_{12} - C_{13} determined by gaschromatography. The results in Table III-VIII show good agreement between the non-acidified sample (a) and the preheated sample (b), with the difference in Hexane Extractable Material corresponding to the increase in free fatty acids.

The gaschromatogram determined on the third sample (c) which was not preheated was very irregular. Time has not yet permitted repetition of the experiment for which reason definite conclusions about the effect of preheating cannot yet be drawn.

TABLE III-VIII

EFFECT OF ACIDIFICATION AND PREHEATING ON EXTRACTION OF SOAPS AS FATTY ACIDS

Extraction Procedure	Hexane Extractables mg/l	Extracted Fatty Acids mg/l	Non-Fatty Acids in Hexane Extract mg/l
a. No HCl added) 1 hr preheating) 3 hrs extraction)	64.0	9.5	54.5
b. 10 ml HCl added) 1 hr preheating) 3 hrs extraction)	80.8	24.5	56.5
c. 10 ml HCl added) no preheating) 4 hrs extraction)	75.2	(36.0)*	(39.0)*

*Results questionable - gaschromatogram very irregular.

Concentration of Individual Free Fatty Acids

The detailed results of the fatty acids determination are presented in Table III-IX. It should be noted here that the concentrations of free fatty acids are of the same order of magnitude as the solubilities of their calcium and magnesium salts.

SUMMARY AND CONCLUSIONS

A liquid-liquid extraction method for the determination of Hexane Extractable Material in sewage has been developed using the direct extraction of a 1-liter sample.

Extraction of fat and fatty acid standards showed an average recovery of 98.5 percent for samples containing from 6 mg to 100 mg fat per liter. The standard deviation of the method, as determined on homogenized raw sewage, was approximately 1.0 mg/l for samples containing from 30 mg/l to 100 mg/l of Hexane Extractable Material, corresponding to a coefficient of variation between three and one percent in this range.

Factors affecting the extraction efficiency, acidification, preheating, duration of extraction, and evaporation of solvent were evaluated, and it is concluded that the most reliable extraction results are obtained when samples are acidified and preheated at 72-75° C for one hour followed by three hours of extraction. The solvent evaporation procedure was found to consist in evaporation to dryness at room temperature, followed by drying for five minutes at 85° C. The final method is described in detail in Appendix A.

Comparison with Other Extraction Methods

Following the development of the extraction method, an analytical program was undertaken in cooperation with the Los Angeles County Sanitation Districts and with the City of Los Angeles. The purpose of this program was to obtain comparison between this new method and present standard methods.

A program of testing was set up with the Los Angeles County Sanitation Districts and conducted at the Joint Water Pollution Control Plant under the direction of Mr. Raymond Stewart.

During the past two years samples of the plant effluent had been analyzed daily by extraction with manual shaking in a separatory funnel as described in Standard Methods (Reference 1, page 185).

It was not possible to conduct any duplicate analysis using both methods due to limitations in available manpower. It was decided instead to use the new extraction method alone for three months and compare the results as reported in Table III-X. Although a rigid statistical comparison is not possible since eventual variations can be due to different analysts and to changes in the plant effluent in addition to true differences in the method, nevertheless the results do not indicate any significant difference between the two methods.

TABLE III-IX

AMOUNT OF FREE FATTY ACIDS IN THE HEXANE EXTRACT OF ACIDIFIED AND NON-ACIDIFIED RAW SEWAGE

Fatty Acid	Sewage Acidified and Preheated Before Extraction	Sewage Preheated But Not Acidified Before Extraction
C_{12}	2.9 mg/l	1.4 mg/l
C_{14}	1.9 mg/l	0.8 mg/l
C_{16}	8.3 mg/l	3.0 mg/l
C_{18}	5.6 mg/l	2.0 mg/l
$C_{18}^{(1x=)}$	5.0 mg/l	2.4 mg/l
$C_{18}^{(2x=)}$	0.9 mg/l	0.3 mg/l

Analysis performed on Wilkens Aerograph 600 D.

Column: 5' x 1/8", 20% FFAP, 70/80 DMCS - W, 267° C.

Detector: Flame, 267° C.

Copier: He 25 Hydrogen 30.

Sample Size: 2.5 µl.

TABLE III-X

COMPARISON BETWEEN THE LIQUID-LIQUID EXTRACTION AND MANUAL EXTRACTION OF "OIL AND GREASE" FROM SEWAGE

Results reported are average of daily analysis for each period

Month	Manual Extraction in Separatory Funnel		Liquid-Liquid Extraction
	1963	1964	1965
June	50.0 mg/l	51.3 mg/l	65.3 mg/l
July	47.1 mg/l	51.9 mg/l	51.7 mg/l
August	50.8 mg/l	54.6 mg/l	46.7 mg/l

The second testing program in cooperation with the City of Los Angeles, Bureau of Sanitation, was conducted at the Hyperion Sewage Treatment Plant under the direction of Mr. Joe Nagano.

Special samples of raw sewage, primary effluent, and digested sludge were collected and analyzed in duplicate using the liquid-liquid extraction method and the standard Soxhlet extraction method (Reference 1, page 292). Four comparisons were made for each type of waste and the results are reported in Table III-XI. There is no significant difference at the 95 percent confidence level between the two methods when used on raw sewage and primary effluent, whereas for digested sludge the Soxhlet extraction gives significantly higher results. The statistical comparison was conducted by pairing the observations and testing the hypothesis that the average difference was not significantly different from zero using the "t" test as described by Dixon (Reference 5).

TABLE III-XI

COMPARISON BETWEEN THE LIQUID - LIQUID EXTRACTION AND SOXHLET EXTRACTION OF "OIL AND GREASE" FROM SEWAGE

Type of Waste	Liquid-Liquid Extraction mg/l	Soxhlet Extraction mg/l		Difference Liquid-Liquid less Soxhlet
Raw Sewage	33.8	31.9		+ 1.9
	60.1	53.8		+ 6.3
	47.7	50.0		- 2.3
	28.9	28.8		+ 0.1
			Avg.	+ 1.5
Primary Effluent	17.8	13.0		+ 4.8
	33.9[1)]	38.6		- 4.7
	33.1	34.2		- 1.1
	8.4	9.6		- 1.2
			Avg.	- 0.5
Digested Sludge	1781.	2295.		- 514.
	670.	890.		- 220.
	560.	790.		- 230.
	1850.	2210.		- 360.
	1940.	2370.		- 430.
			Avg.	- 351.

[1)] Emulsion formed - too fast mixing.

IV. QUANTITATIVE DETERMINATION OF FLOATABLE MATERIAL IN DOMESTIC SEWAGE

INTRODUCTION

Before any treatment methods for removal of floatables could be investigated and evaluated, it was necessary to develop a usable laboratory method for the determination of floatable material in both raw and treated wastes, as already pointed out by Pearson (Reference 6). The method should be able to measure the amount of floatable material both in a waste and also the amounts of material which would float after a waste had been discharged into a receiving water with a density and ion concentration different from that of the waste.

Ideally, the method should also include any "potential floatable material" in a waste water, i.e., any floatable material which may not be floating at the time of discharge but which may be liberated with time; for example, grease particles adsorbed to settleable solids may be desorbed in the marine environment and subsequently rise to the ocean surface as described by Ludwig (Reference 7). Since this effect will depend upon both the individual wastes and the conditions in the actual disposal area (for example whether or not sludge banks are formed) it was not considered feasible to develop a potential floatables material test at this time.

REVIEW OF PREVIOUS WORK

Very little information was found in the literature regarding the determination of floatable material and no information appeared which dealt with the concept of floatable matter as related to the density for a receiving water.

One method proposed by Pomeroy (Reference 8) used a straight glass cylinder as flotation vessel. After a given flotation period, the cylinder was drained carefully, stopping before any floating material reached the bottom stop-cock. The flotation cylinder was then washed with chloroform, the chloroform transferred to a preweighed flask and evaporated, leaving the floatable oil and grease as residue. Non-soluble floatable matter was not measured by this method.

A seond test was developed by Nusbaum (Reference 9a) and recently modified (Reference 9b). This method is based upon measuring the floatable material as the amount of filterable solids in the top three centimeters of sewage after a one-hour settling period in a standard cylinder, but the method will result in inaccurate results when used on wastes with a high concentration of suspended solids compared to the concentration of floatable material.

Neither of these methods appeared suitable for this project, and a method was therefore developed which would measure total floatables.

THEORETICAL CONSIDERATIONS

For the purpose of evaluating treatment methods with respect to the removal of floatable material, it was considered necessary to develop a test which would not only determine the floating "oil and grease" but rather measure the total amount of floatable material in a given sample. The main objective of the removal of floatable material is to avoid an aesthetic nuisance, and, therefore, one approach would be a measurement involving evaluation of the aesthetic affects (as by areal color photography). A second approach, used in this study, is based on measuring the total mass of the floatable material. This approach should be valid for most wastes except perhaps those industrial wastes that contain a significant amount of hydrocarbons capable of spreading as a thin film over a very large area.

Factors Affecting Amount of Floatable Material

Of the factors which might affect the amount of floatable material in sewage as determined by a test procedure, the most important was believed to be the time of flotation, i.e., the time during which the particles with densities less than the surrounding medium could rise to the surface. The second most important factor was believed to be the effect of mixing; previous experience described by Pomeroy had shown that even slight differences in the handling of a sample after collection could result in large differences in the amount of floatable material (Reference 8). In order to obtain more reproducible results, it was therefore decided to mix all samples in a standardized manner before flotation. Temperature may also be important, and the tests should always be conducted at the same temperature as that of the receiving body of water.

The purpose of this project being to develop improved treatment methods related to ocean disposal, the third important factor which should be considered was the effect of sea water on the amount of floatables, or in other words: Is this additional amount of floatable material created after contact with sea water so significant in domestic sewage as to warrant the development of a special test?

Two effects of the sea water were possible, the first being the density effect and the second the effect of the high concentration of divalent ions in sea water which might affect surface properties of suspended material. Considering the concentrations of Ca^{++} and Mg^{++} in waste water, 100 mg/l - 1000 mg/l, concentrations of fatty acids, 10 mg/l - 100 mg/l, and the solubility of the calcium and magnesium salts of the palmetic acid and stearic acid, 20 mg/l - 40 mg/l, additional precipitation of soaps from contact between domestic waste and sea water was believed unlikely.

EXPERIMENTAL WORK

Apparatus

Several different types of flotation cylinders were tried out in the initial phase of the program, and it was found that the best and most practical instrument to use in the separation of floatables from sewage was a three-liter funnel provided with a steep conical bottom (Imhoff Cone) and with a bottom stop-cock with a seven millimeter bore. The funnel was provided with a three-liter mark, a 200 milliliter mark, a 40 milliliter mark, and a 10 milliliter mark. In addition, provisions were made for mixing of the content be means of a paddle with adjustable speed. Funnel, mixer, and all surfaces which came in contact with the samples were coated with Teflon to prevent grease from sticking to the surfaces. The set-up, including six Flotation Funnels, is shown in Figure IV-1, and a detailed description is given in Appendix B.

Outline of Experimental Work

1. The first part of the experimental work consisted in a preliminary evaluation of the main factors which might affect the amounts of floatable material in a given waste, followed by the selection of the optimal floatables determination procedures on the basis of this evaluation.

2. The primary evaluation was then followed by the testing of the selected procedure on several different types of sewage. This testing resulted in two slight modifications warranted by the experience gained from work with wastes containing high concentrations of suspended and settleable solids and low concentrations of floatable material.

3. Finally, the precision and accuracy of the selected methods were determined for different concentrations of floatable material.

The final procedure is described in detail in Appendix B.

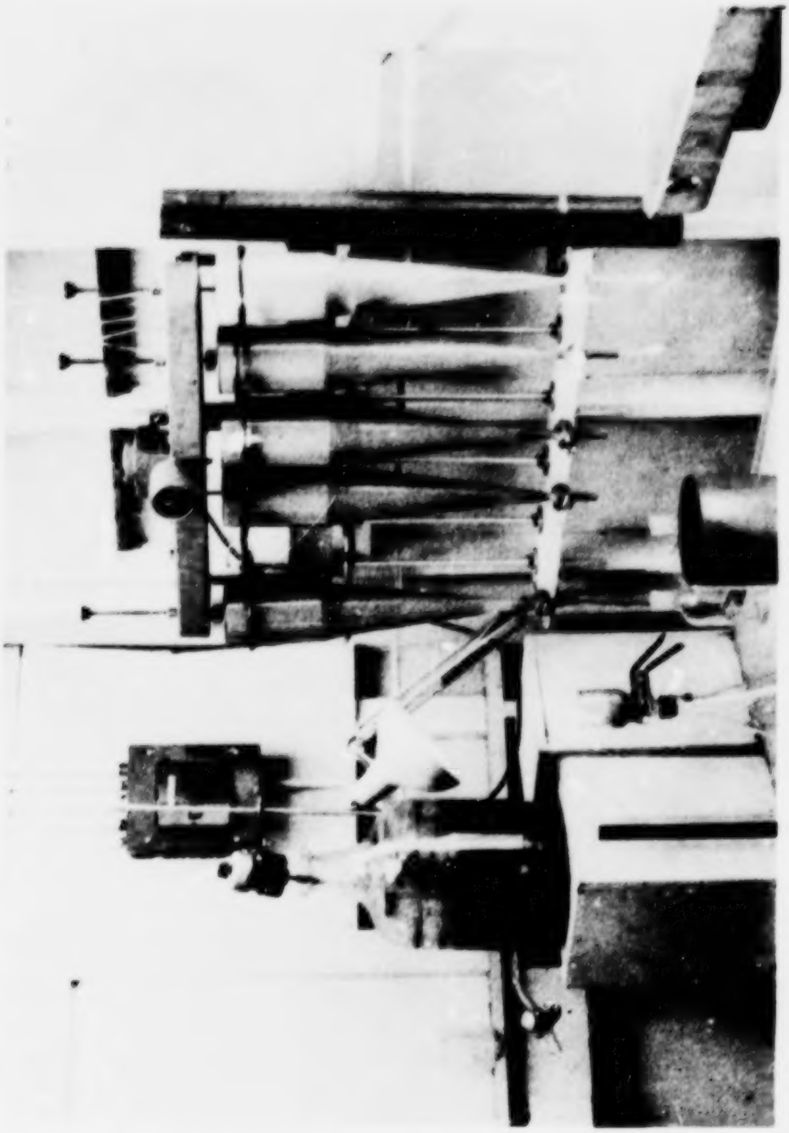
Preliminary Evaluation

On the basis of the theoretical considerations above, it was decided to investigate the effect of mixing time, flotation time, and the effect of sea water on the amount of floatable material. The three factors were investigated on two different types of sewage in a 3 x 3 x 2 factorial experiment for each of the two sewages. Mixing and flotation were each investigated at three levels, 15, 30 and 60 minutes, and the effect of sea-water was investigated by comparison between an "infinite" sea water dilution and no dilution with sea water. The "infinite" sea water dilution was obtained by diluting one volume of the sewage to be tested with one volume of filtered sea water to which 26.5 g NaCl, 2.5 g $MgCl_2$, 3.3 g $MgSO_4$, and 1.2 g $CaCl_2$ had been added per liter of sea water. (Formula for artificial sea water Reference 10). This salt mixture was used in the two factorial experiments. Later, when the effect of salt addition was found to be small, 35 grams of analytical grade sodium chloride was added per liter instead. The final mixture then had approximately the same density and ion concentration as an infinite dilution.

Six Flotation Funnels were available and were placed in a rack provided with a standard mechanical stirrer as used in coagulation tests, Figure IV-1. The limited number of available funnels restricted the block size to six tests, and subsequently a design described by Cochran and Cox (Reference 11) was used. Two replications each with 3 blocks, or a total of 36 tests, were made on each of the two types of raw sewage investigated. The two types of sewage were chosen from local treatment plants to represent low and high concentrations of floatable material, respectively.

The experimental layout together with the treatment codes used in table of results is presented in Table IV-I. Tables IV-II and IV-III give the six blocks used with the two types of sewage.

FIGURE IV- 1



FLOTATION FUNNELS AND MIXING UNIT

TABLE IV-I

EXPERIMENTAL LAY-OUT AND TREATMENT CODES

Factor	Unit	Level Code: 0*)	1*)	2*)
Mixing at 40 RPM	Minutes	15	30	60
Flotation	Minutes	15	30	60
Sea Water Addition	Amount of Dilution	"00"	None	

*) The numbers 0, 1, and 2 designate the level of each factor in the treatment code. The treatment code consists of three numbers in a parenthesis, where the first number designate the level of mixing, the second the level of flotation, and the third the amount of sea water dilution. For example, (210) means 60 minutes of mixing followed by 30 minutes of flotation on a sample with no sea water addition.

Each block of experiments (six tests) was carried out in the following manner: An 18-liter sample of raw domestic sewage was collected in a 5-gallon carboy (provided with a one-inch bottom outlet) and transported to the laboratory. The content of the carboy was mixed with a propeller mixer to insure as uniform a distribution of the particulate matter as possible. The six Flotation Funnels were then filled from the bottom outlet of the carboy in a random order while the content was being mixed. Sea water-salt was added to those funnels which were scheduled in the experimental design to receive this treatment. The individual samples were mixed and subsequently allowed to float for the time intervals specified in the experimental layout shown in Tables IV-II and IV-III. After the end of the specified flotation time, the stop-cock was opened full for a very short time, thus washing all sludge collected in the bottom of the Flotation Funnel out and loosening any sludge which might stick to the sides of the Teflon coated funnels. The flow was then reduced and a total of 2.8 liters out of the 3 liters wasted over a period of 5 minutes. The stop-cock was closed and the remainder 200 ml left for 15 minutes, allowing any floatables which might have been caught in the downflowing liquid to rise to the surface. After 15 minutes, an additional 160 milliliters were wasted (down to the 40 ml mark on the cone) and the remainder of the sample left for an additional 5 minutes after which all but the last 10 milliliters was wasted. A Teflon coated Millipore filter holder (Millipore Catalog No. 10 047 00) with a preweighed glass fiber filter was placed underneath the funnel and the last 10 milliliter of sample containing all floatable material filtered. The inside of the funnel was washed with distilled water from a plastic squeeze bottle, taking care to wash all particles hanging on the walls down on the filter paper. Finally, the filter paper was washed with 100 ml distilled water, the sides of the filter holder cleaned with a water jet from the squeeze bottle and the filter sucked dry. The filters with the floatables were dried in an oven at 40° C to constant weight.

TABLE IV-II

EFFECT OF MIXING, FLOTATION, AND SEA WATER ADDITION ON AMOUNT OF FLOATABLE MATERIAL IN SEWAGE WITH LOW CONCENTRATION OF FLOATABLES

Replicate I

Block a		Block b		Block c	
Code	mg/l	Code	mg/l	Code	mg/l
(100)	9.8	(200)	1.4	(000)	5.5
(210)	9.9	(010)	4.3	(110)	4.7
(020)	9.7	(120)	5.0	(220)	6.4
(201)	3.9	(001)	2.1	(101)	4.4
(011)	5.7	(111)	1.7	(211)	3.5
(121)	5.4	(221)	1.9	(021)	5.0

Replicate II

Block a		Block b		Block c	
Code	mg/l	Code	mg/l	Code	mg/l
(200)	21.9	(000)	14.5	(100)	11.9
(110)	23.9	(210)	5.2	(010)	6.9
(020)	13.2	(120)	11.5	(220)	9.9
(101)	6.9	(201)	2.6	(001)	6.0
(011)	14.7	(111)	7.1	(211)	13.3
(221)	11.3	(021)	4.9	(121)	7.7

Analysis of Variance
(For details see Appendix D)

Source of Variance	Degree of Freedom	Sum of Squares	Mean Sum of Squares
Blocks and Replications	5	557	111
Mixing	2	7	3
Flotation	2	8	4
Sea Water	1	131	131**
Mixing - Sea Water	2	18	9
Flotation - Sea Water	2	43	22*
Mixing - Flotation, Third Order Interaction and Error	21	98	4
Total	35	862	

**Significant at 1% level.

*Significant at 5% level.

TABLE IV-III

EFFECT OF MIXING, FLOTATION, AND SEA WATER ADDITION ON AMOUNT OF FLOATABLE MATERIAL IN SEWAGE WITH HIGH CONCENTRATION OF FLOATABLES

Replicate I

Block a		Block b		Block c	
Code	mg/l	Code	mg/l	Code	mg/l
(200)	36.5	(000)	21.5	(100)	4.6
(010)	37.3	(110)	12.9	(210)	24.9
(120)	31.2	(220)	18.7	(020)	8.7
(101)	32.3	(201)	62.0	(001)	13.4
(211)	27.4	(011)	17.3	(111)	12.7
(021)	40.9	(121)	12.8	(221)	22.4

Replicate II

Block a		Block b		Block c	
Code	mg/l	Code	mg/l	Code	mg/l
(100)	19.1	(200)	16.1	(000)	25.4
(010)	19.0	(110)	24.6	(210)	29.3
(220)	15.6	(020)	16.7	(120)	24.1
(201)	13.9	(001)	17.5	(101)	25.1
(111)	8.3	(211)	14.1	(011)	13.1
(021)	12.9	(121)	13.6	(221)	19.3

Analysis of Variance
(For Details see Appendix D)

Source of Variance	Degree of Freedom	Sum of Squares	Mean Sum of Squares
Blocks and Replications	5	1,712	342
Mixing	2	137	68
Flotation	2	65	33
Sea Water	1	1	1
Mixing - Sea Water	2	52	26
Flotation - Sea Water	2	395	197
Mixing - Flotation, Third Order Interaction and Error	21'	1,671	80
Total	35	4,033	

The results are reported in Tables IV-II and IV-III for the two types of sewage investigated. The following conclusions can be drawn on the basis the two analyses of variance: The effect of sea water was significant at the 99 percent confidence level for the sewage with the low floatables concentration, whereas no significant effect was found with the sewage containing the higher concentration of floatables. Of the second order interactions, only the Flotation - Sea water effect was significant at the 95 percent confidence level, and again only for the sewage with the low floatables concentration. (An experiment described in detail in Chapter VII showed that there would be a significant decrease in the amount of floatable material if flotation times of five minutes or less were used.)

The difference in the significance of sea water addition in the two cases is likely due to the lower level of homogeneity of the sewage with the high concentration of floatable material, which will mask a numerically small sea water effect.

During these experiments, there had been occasional difficulties with the high blanks; in some cases the 100 ml washing would not remove the salts completely from the glass fiber filters, and it was believed possible that the significant sea water effect might have been caused by salts adsorbed on the glass fiber filter. It was therefore decided to repeat part of the experiment on a sewage containing a low concentration of floatable material, and collecting the floatables on pre-weighed membrane filters instead of glass fiber filters. (The membrane filter had been tested and showed no tendency of adsorbing salts from the sea water.) The experiment was set up in 10 blocks of six tests each, three of these were diluted "infinitely" with sea water as described above and the remainder three received no sea water dilution. All samples were mixed for 15 minutes at 40 RPM and for 1 minute at 100 RPM followed by flotation for 30 minutes. The results are presented in Table IV-IV and show that the sea water addition is significant at the 99 percent confidence level. The average concentration of floatable material was 1.1 mg/l without sea water addition and 1.8 mg/l with sea water addition. This corresponds to an average concentration of potential floatable material of 0.7 mg/l in this sewage.

Conclusions to the Preliminary Evaluation: Of the factors and levels investigated, only the addition of sea water proved significant and this probably only for wastes containing small amounts of floatable material. Thus, contact between sea water and domestic waste water does cause more solids to float to the surface, and although the amount is small for the two wastes used for this study, the possible effect of sea water should be evaluated for each particular waste intended for disposal into the marine environment.

Since there were no differences between the different mixing and flotation periods investigated, it was decided to use 30-minute periods of flotation because of practical reasons (manipulation was more convenient when several tests were run at the same time). Two procedures were then set up, differing only in the sea water-salt addition step for the determination of floatable material.

Testing and Modification of Selected Procedures

The procedures as selected above were used to determine the amount of floatable material in samples of both raw sewage and primary effluents

TABLE IV-IV

ADDITIONAL INVESTIGATION OF THE EFFECT OF SEA WATER ON AMOUNT OF FLOATABLE MATERIAL IN SEWAGE WITH LOW CONCENTRATIONS OF FLOATABLES

(All blocks received same treatment)

Treatment Code	Block b mg/1	Block c mg/1	Block d mg/1	Block e mg/1	Block g mg/1
(110)	1.5	3.7	1.2	1.6	0.6
(110)	1.5	2.3	1.2	1.3	0.2
(110)	0.9	2.8	1.1	0.9	0.3
(111)	0.8	1.4	0.7	0.6	0.1
(111)	1.1	2.2	0.7	0.6	2.0
(111)	1.6	2.9	0.4	0.2	0.6

Treatment Code	Block h mg/1	Block i mg/1	Block j mg/1	Block k mg/1	Block l mg/1
(110)	3.8	3.1	2.7	1.4	0.8
(110)	3.8	2.5	1.9	2.0	1.0
(110)	3.8	2.7	1.4	1.1	0.9
(111)	0.8	1.5	0.9	1.5	0.4
(111)	3.2	1.1	1.0	0.9	0.4
(111)	2.8	0.9	1.0	0.3	0.3

Analysis of Variance

Source of Variance	Degrees of Freedom	Sum of Squares	Mean Sum of Squares
Blocks	5	36	7
Sea Water Effect	1	7	7**
Error	23	16	0.7
	29	60	

**Significant at 1% level.

containing from 50 mg floatable material per liter to 1 mg floatable material per liter. The highest concentration of floatable material was prepared artificially by adding skimmings from a primary settling tank to samples of raw sewage. During the course of these experiments two interferences were observed which adversely affected the determination of floatable material.

When samples containing large amounts of settleable solids were analyzed, it was noted that the settleable solids would tend to trap otherwise floating particles, resulting in low recoveries. This error was greatly reduced by changing the mixing procedure as follows: The sample was mixed at 40 RPM in the Flotation Funnel for 15 minutes (instead of 30 minutes) followed by a 5-minute settling period during which floatable and settleable material separated partly. The settling was then followed by a rapid mixing at 100 RPM for 1 minute to liberate floatable material caught in the settled solids. This second mixing period was chosen such that the floatables already on the surface would not again be brought into contact with the settleable material. This second mixing period was followed by the 30-minute flotation period and discharged as described above. Visual inspection showed a marked decrease in the amount of material floating on the surface of the liquid discharged from the test cylinder when this modification was used.

The second interference was connected with the presence of large amounts of suspended solids in samples containing only small amounts of floatable material. A sample which contains 100 mg/l suspended solids and 1 mg/l floatable material will contain 1 mg suspended solids (in 10 ml) and 3 mg floatables (from the three liters) in the last 10 ml to be filtered.

It was first tried to correct for this error by filtering the volume from the 40 ml mark to the 10 ml mark through a separate preweighed filter, calculate the amount of suspended solids per 10 milliliters and subtract from the final weight. Although this procedure worked well, it was time-consuming, and instead it was decided to "dilute" the suspended solids. The samples were mixed and settled as above until the last 10 ml were left. Then 500 ml distilled water (or 500 ml filtered sea water if sea water addition is used) was added slowly along the sides of the Flotation Funnel and the mixture was allowed to settle again for 15 minutes before being discharged in steps to the final volume of 10 milliliters. This procedure resulted in a 50 fold dilution of the suspended solids without any loss of floatable material. Analysis of five samples of raw sewage using this modification resulted in suspended solids corrections of less than 0.1 mg for all five samples containing an average of 1.0 mg floatables per liter. This modification was therefore adopted for the final procedure.

It was noted that settleable solids at times tended to clog the stopcock; this can easily be corrected for by using a thin Teflon coated brass rod which can be inserted from the top of the cylinder to clean the stopcock. Also at times grease particles tended to stick to the bottom of the Millipore filter holder; this can be corrected by weighing a small extra piece of filter paper with the filter and use this small piece (held with a pair of forceps) to wipe the filter holder clean; the small piece is then added to the large filter and both weighed as before.

Precision and Accuracy of Final Procedure

The modifications were incorporated into the final procedure which is described in detail in Appendix B. Following this, the precision of the method was determined from replicate analysis on three different types of sewage. The accuracy of the method was estimated at the same time, using the following method: For each type of sewage, the precision was determined from five replicates and the waste from these five tests was collected in one large polyethylene container and allowed to settle overnight. The next morning, the bottom layer (about 13 liters) was siphoned off and the remainder, including any floating material, transferred quantitatively to a Flotation Funnel and the amount of floating material determined. The percent recovery was calculated as follows:

$$\text{Percent Recovery} = \frac{\Sigma \text{ Floatables in the five tests}}{\Sigma \text{ Floatables in the five tests + Floatables collected in the second determination}} \times 100$$

The waste from the second floatables determination was subsequently inspected visually for floating material, but in neither of the tests were any floatable material seen.

Determinations were made on three different types of sewage; the results are reported in Table IV-V and show the coefficient of variation to vary from 5.7 percent for a sewage containing 49 mg/l floatable material to 20 percent for 1.0 mg/l floatable material.

TABLE IV-V

PRECISION AND ACCURACY OF THE FLOATABLES DETERMINATION METHOD

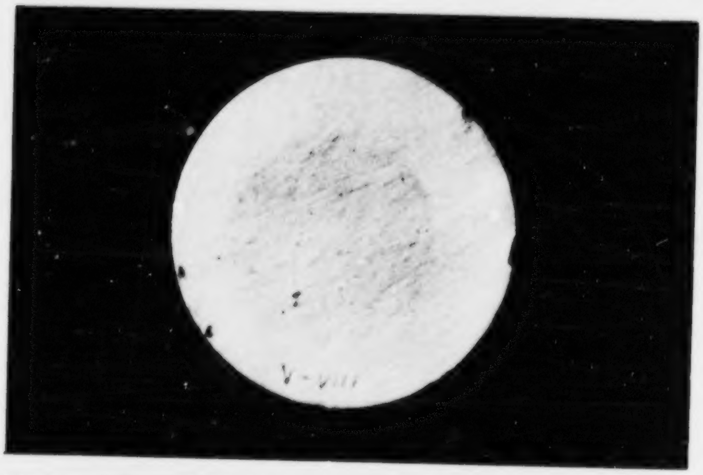
Type of Sewage	Avg. Floatables Concentration mg/l	No. of Samples	Coef. of Variation	Percent Recovery
Raw*	49. mg/l	5	5.7%	96%
Raw	1.0 mg/l	5	20%	92%
Primary Effluent	2.7 mg/l	5	15%	91%

*)Additional floatable material added from skimmings from a primary tank.

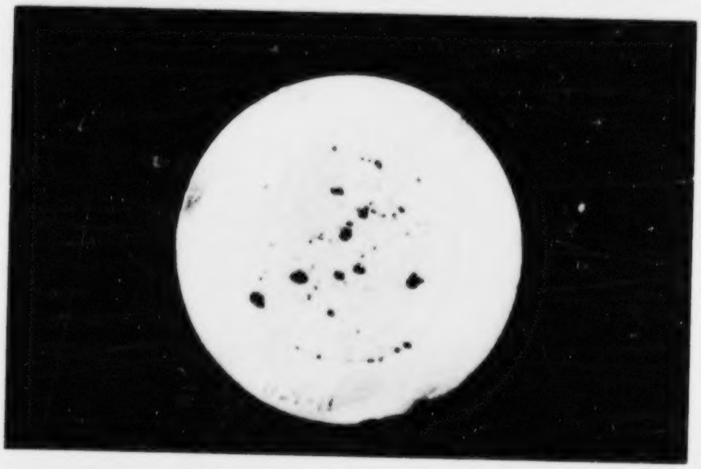
Figure IV-2 shows typical examples of floatables colleced from three liter samples of different types of raw sewage.

FIGURE IV-2

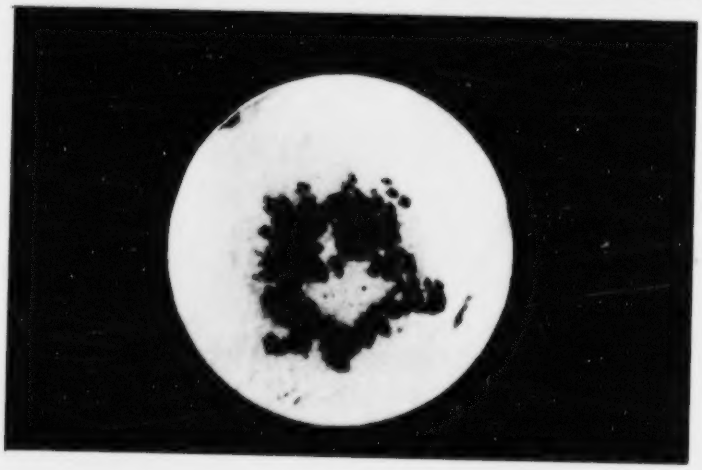
TYPICAL EXAMPLES OF FLOATABLES COLLECTED



0.5 mg/l
FLOATABLES



10.0 mg/l
FLOATABLES



100.0 mg/l
FLOATABLES

SUMMARY AND CONCLUSIONS TO CHAPTER IV

1. A method for determination of floatable material in domestic sewage has been developed using a special Teflon coated Flotation Funnel. The method includes material which float only after contact with sea water.

2. The effects of mixing time, flotation time, and sea water addition were investigated in the following levels:

   Mixing: 15, 30, and 60 minutes.

   Flotation: 15, 30, and 60 minutes.

   Sea Water Dilution: No dilution and "infinite" dilution.

   Only sea water dilution was found to be significant, and this only for wastes containing low concentrations of floatable material.

3. The composition of the potential floatable material has not been determined; this is being considered for the third project year.

4. The coefficient of variation and accuracy of the test developed varied from 5.7 percent and 96 percent recovery of floatable material for a sewage with 49 mg floatable material per liter to 20 percent and 92 percent recovery for sewage with 1.0 mg floatable material per liter.

V. LABORATORY EVALUATION OF PROPOSED FLOATABLES REMOVAL METHODS

INTRODUCTION

The objective of this part of the study was to evaluate on a laboratory scale and bench scale several schemes aimed at a high degree of removal of floatable material from domestic sewage.

The possibilities of biological conversion of floatable material either into sinkable material by adsorption onto activated settleable sludge or into soluble material by metabolism were investigated first, but unfortunately neither a stable adsorption nor an effective metabolism could be achieved.

Subsequent studies were therefore directed toward evaluation and improvement of physical methods for the removal of floatable material. These physical methods studied included fine bubble aeration, sea water addition, prolonged time of flotation, and pressure aeration.

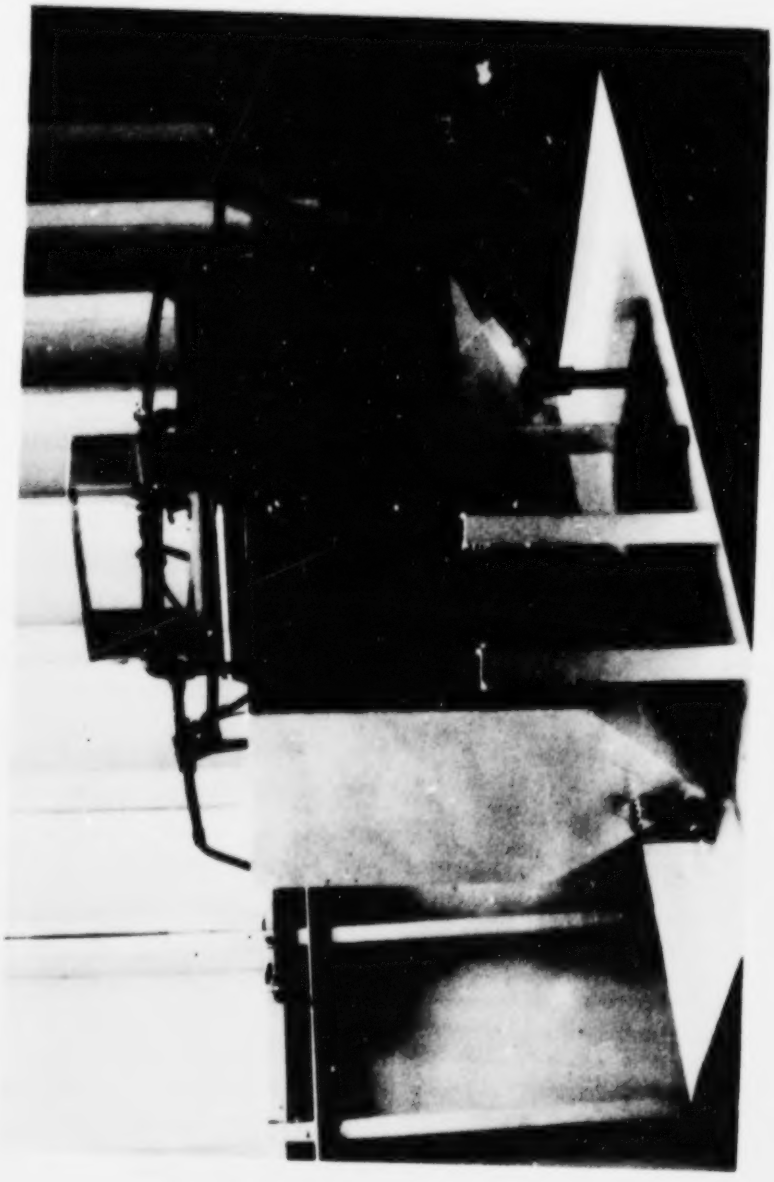
BIOLOGICAL CONVERSION OF FLOATABLE MATERIAL

The mechanism of biological flocculation has been discussed by McKinney (Reference 12). During biological flocculation, the influent is intimately mixed with activated sludge. Direct collision between the activated sludge particles cause these particles to agglomerate and form a large readily settleable floc. In this process, the grease particles may be rendered settleable by adsorption on the floc, and, upon settling, are removed from the sewage. When the activated sludge is in the endogenous respiration phase, the floc is characterized by a very high adsorptive capacity for colloidal particles. These sludge characteristics were utilized in the first phase of the biological investigation.

Floatables Adsorption

The flow diagram of the first bench scale unit designed to test biological adsorption is shown in Figures V-1 and V-2. Chamber A acted as the adsorption chamber where activated sludge in the endogenous phase (returned from Chamber D) was intimately mixed with the incoming raw sewage by means of air. The mixture then flowed over the top of the baffle into the Chamber B where the separation of settleables and floatables took place. The combined detention time was initially 2.5 hours intended to be decreased when stable conditions were obtained. Settled material with adsorbed floatable material was removed with the effluent, and the remaining floatable material transported to the end of the tank by water jets. A rolling scraper E transferred this floatable material together with a small amount of the influent (as a nutrient source) to the Chamber C for further treatment. Chamber C was separated from Chamber D by a baffle wall and floatable material was not able to leave Chamber C before being converted into suspended or settleable material by adsorption. The purpose of Chamber D was to give the sludge which passed under the baffle additional aeration before it was recirculated back to Chamber A. The detention time of Chamber C varied between 7.5 hours and 12 hours total.

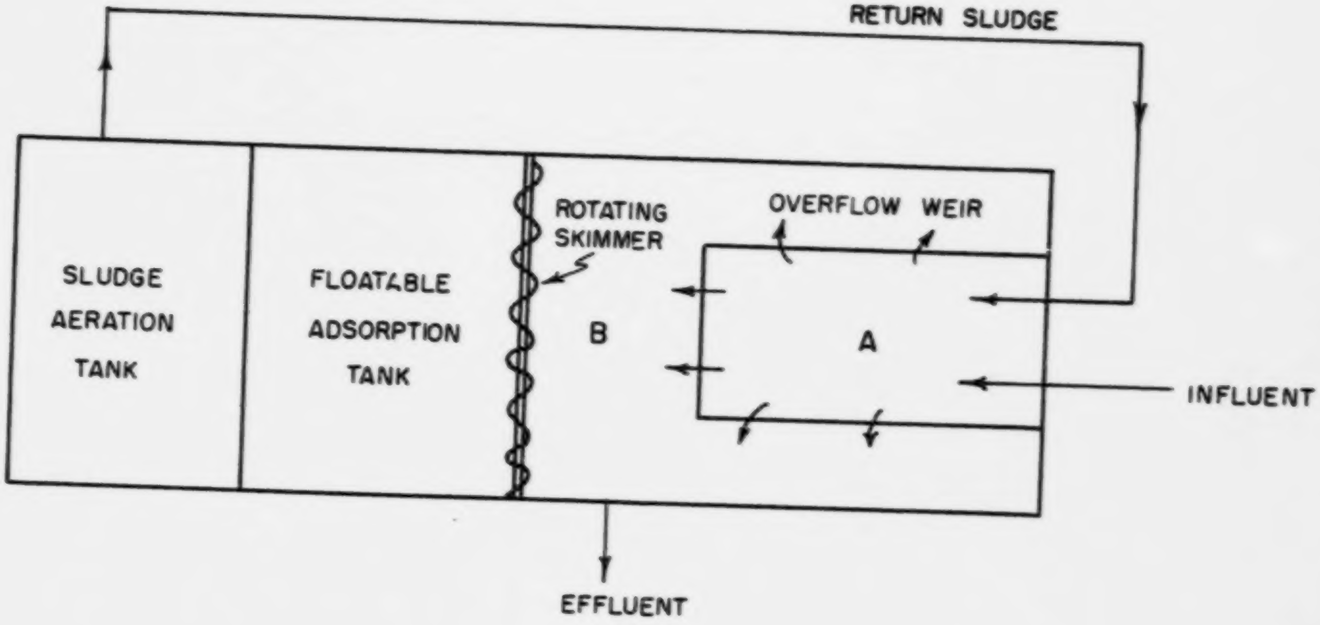
FIGURE V-1



BENCH SCALE UNIT FOR FLOATABLES ADSORPTION

FIGURE V-2

FLOW DIAGRAM OF FLOATABLES ADSORPTION UNIT


RETURN SLUDGE
ROTATING SKIMMER
OVERFLOW WEIR
SLUDGE AERATION TANK
FLOATABLE ADSORPTION TANK
B
A
INFLUENT
EFFLUENT

A - AERATION TANK
B - SETTLING TANK

Thus, any floatable material in the influent to the plant would be unable to leave the plant until such time as it either became enmeshed in other material (the total mass being settleable), or became settleable itself. Even if the material found its way from Chamber C to D before it was fully settleable, and was then recirculated back to Chamber A, this material would eventually go back to Chamber C without being discharged in the plant effluent. In order to maintain constant influent characteristics, synthetic sewage with added real floatables was used as the substrate in testing the pilot plant operation. The formula for this sewage is given in Table V-I.

TABLE V-I

COMPOSITION OF SYNTHETIC SEWAGE

	Composition of Stock Solutions (gr/1)	Composition of Synthetic Sewage (mg/1) *)
Solution 1		
KCl	2.79	14.0
NH_2CONH_2	12.0	60.0
$NaH_2PO_4 \cdot H_2O$	10.0	50.0
$Al_2(SO_4)_3$	20.0	100.0
Fuller's Earth	3.34	16.7
Starch, Soluble	40.0	200.0
Solution 2		
Castile Soap	20.0	100.0
Beef Extract	15.0	75.0
Crisco	20.0	100.0
ABS, 70%	1.64	8.2

*) The two stock solutions were added to water at the rate of about 5 ml/l each to produce the final synthetic sewage.

The two solutions were added to water at the rate of about 5 ml/l each; the resulting solution approximated the strength of domestic sewage.

Throughout most of the study sewage floatables were added directly to Chamber A on a batch schedule once a day. This material was obtained at the Richmond, California Water Pollution Control Plant and was added, after comminution, at a rate proportional to that of the Richmond plant. These floatables, like those found in nearly every plant, contained substances other than grease and oils, mainly other organic debris such as hair, wood fragments, plastics, paper, and substances that are heavier than water but are enmeshed in materials that are lighter than water.

During the testing to be reported, the suspended solids concentration in Chambers A and B was kept between 1,000 and 2,000 mg/l, and the suspended solids concentration in Chambers C and D between 3,000 and 4,000 mg/l. Although the suspended matter remained quite high in the system as shown by the suspended solids figures quoted above, there was no indication as to the percentage of these solids which could be considered to be the active mass. Microscopic examinations of the solids revealed significant numbers of protozoa present, although the solids portion did not appear to resemble "good" activated sludge, but looked more like agglomerated grease particles. Grease particles of colloidal size were also very abundant in the activated sludge under microscopic examination.

Results of the Floatables Adsorption Test: The operation of this treatment unit revealed that some of the floatable material was indeed converted into settleable material by adsorption, but that subsequent pumping or other handling will result in partial desorption. Visual inspection of the treatment unit also showed that a large percent of the grease collected on the walls and other parts of the unit and was not being rendered settleable nor soluble. Microscopic examinations confirmed that these finds, as agglomerated grease was seen in the aeration unit sludge (Chambers C and D), and this material was of such a nature that it easily clung to the walls of the tanks. After a period of grease build-up on the walls and pipes, some sloughing was noted, at which times the amount of floating grease particles became very evident in the effluent.

Because of these difficulties with floatables build-up and wall effects, quantitative evaluation of the performance of the test unit was not possible. It was observed that this treatment scheme did not provide sufficient quantities of substrate which could be utilized for sludge growth because only small amounts of substrate were able to flow over the weir between Chambers B and C for use by the organisms in the activated sludge. Abundant grease and other floatable materials were present but because of their resistance to biological degradation they did not serve as an adequate metabolic substrate for the biota present. In addition, floatables determination on the effluent showed that some floatable material escaped from the unit with the effluent. It was found that this floatable material was actually adsorbed to settleable sludge in the unit but that the turbulence in the effluent line and the mixing during the sampling and testing caused the floatable material to separate from the sludge and rise to surface again.

The conclusions based upon the results of this experiment were that adsorption of floatable material onto settleable sludge was not a significant improvement, as far as the effluent quality was concerned, over the existing methods of floatables removal.

Floatables Metabolism

Based upon the above conclusions, a second system was designed to take advantage of the adsorptive capacity of activated sludge as well as the metabolic activity of the organisms in the sludge. The major difference between this system and an activated sludge plant of conventional design lies in the methods by which the sludge and the surface skimmings are handled. Since the reduction of organic material, expressed as BOD, is of little concern as long as the material is being properly discharged into the marine environment, all excess sludge that was capable of settling was discharged. This resulted in an effluent nearly equivalent to the strength of a raw sewage, the difference being in the form of the material discharged. Enough sludge to keep the unit active was recirculated back to the aeration tank. The flow diagram is shown in Figure V-3.

The flow from the aeration tank was taken to the conically shaped sedimentation tank, entering the tank at about mid-depth. A provision for skimming the surface was incorporated and was used on a manual basis (about twice per day) whereby the floatable matter could be returned to the aeration tank.

The pilot unit was operated for short aeration times to determine the effectiveness of floatable removal with aeration and sedimentation only. The start-up of the pilot unit is represented as Day 1 in the tabular summary of results presented as Table V-II.

The unit was started using a dog food mixture (about 300 mg/l COD) for 30 hours, whereafter additional grease was added to this synthetic feed. This additional grease and fat fraction was made from 60 percent Crisco and 40 percent Castile Soap. The materials were blended (by a Waring Blender) with the concentrated dog meal, and added at a constant rate. However, during the periods between feed make-up some of the grease separated from the feed and floated on the surface of the feed reservoir, therefore causing lower values of applied hexane extractable material. This is apparent from the feed grease concentrations recorded in Table V-II. The highest values (71 mg/l) were obtained on those days when no floating material was visible on the feed reservoir.

Sampling and Analyses: Grease analyses were carried out by liquid-liquid hexane extraction on the feed (as it entered the aeration tank), on the sedimentation tank effluent, and on the contents of the aeration chamber (mixed liquor). The sampling procedure consisted of thoroughly scraping the walls of the aeration tank, resuspending materials which clung to these surfaces, and transferring all material floating on the sedimentation tank to the aeration tank, after which the air supply was shut off and the surface grease was sampled in proportion to the total sample. The remainder of the grease sample was taken from the mixed liquor after aeration was commenced. This latter portion did not include the floatable portion. Thus, the sampling method attempted to get a representative sample containing relative portions of floating and suspended materials from the mixed liquor.

The recirculated sludge was withdrawn from the base of the conically shaped sedimentation tank, and the effluent draw-off point was at approximately mid-depth, while the influent was at the two-thirds depth. This arrangement allowed all floatable materials to accumulate on the sedimentation tank surface, where they were collected manually twice a day and returned to the aeration tank.

Some difficulties arose with the pumping of the concentrated synthetic sewage, but fortunately all such circumstances took place at times when these failures could be recorded. These failures are evident in the results

FLOW DIAGRAM OF FLOATABLES METABOLISM UNIT

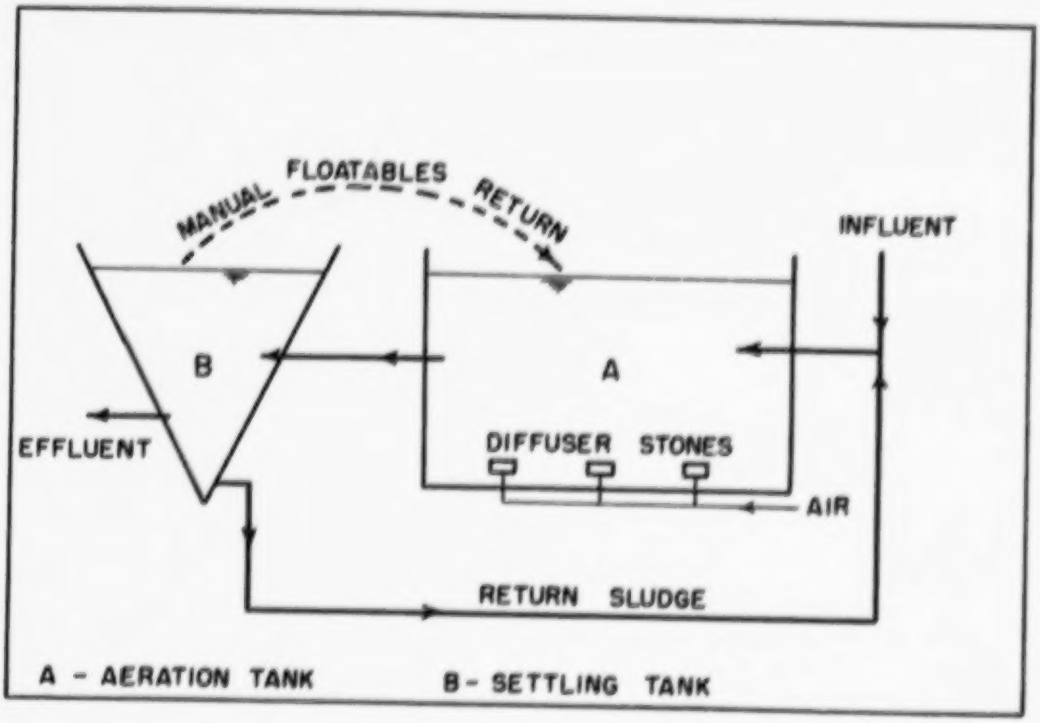

MANUAL FLOATABLES RETURN
INFLUENT
B
A
EFFLUENT
DIFFUSER STONES
AIR
RETURN SLUDGE
A - AERATION TANK
B - SETTLING TANK

TABLE V-II

RESULTS OF PILOT PLANT INVESTIGATION DURING SHORT DETENTION[1] PERIODS

							Grease Results						
							Concentrations			Accumulated Values			
Day No.	Infl. Flow 1/hr	Recirculation %	Detention[1] Time in Aerator hr	M.L.S.S. mg/1	Effl.S.S. mg/1	SVI	Feed mg/1	Effl. mg/1	M.L. mg/1	Feed g	M.L. g	Sed. Tank g	Effl. g
1	17.5	-	1.88	895	-	190	-	-	-	-	-	-	-
2	15.6	53.0	2.12	672	-	178	Grease added to synthetic sewage						
3	25.8	30.2	1.28	1456	62	110	27.0	14.1	207.4	16.7	6.85	0.36	8.8
4	25.5	27.6	1.29	1388	138	151	71.0	7.3	500.4	35.0	16.6	0.19	13.1
5	25.0	-	1.32	-	-	-	71.0[2]	20.[2]	-	38.7[2]	-	-	25.5[2]
6	24.5	-	1.35	-	-	-	40.0[2]	40.[2]	-	63.7[2]	-	-	50.5[2]
7	24.0	25.0	1.37	1660	168	87.5	57.1	43.4	1079.0	93.9	35.8	1.13	72.3
8	28.8	20.8	1.15	574	152	69.8	29.0	48.4	758.	113.9	25.0	1.26	105.7
9	27.6	17.6	1.20	624	176	80.1	45.2	16.2	533.	136.3	17.6	0.42	113.4

(1) Detention time = Aerator Volume ÷ influent flow.

(2) Values assumed on basis of previous data and observations of the pilot unit.

$$\text{Experiment Grease Recovery} = \frac{(\Sigma\ \text{Effl. Grease} + \text{M.L. Grease @ day 9} + \text{Sed. Tank Grease @ day 9})}{\text{Grease Feed During Experiment}} \times 100$$

$$= \frac{(113.4 + 17.6 + 0.42)\ 100}{136.3} = 96.5\%$$

of Table V-II where only small quantities of added grease are recorded for a daily interval (Day 5 for example).

Discussion of Results: Table V-II summarizes all important data obtained during the duration of this experiment. The influent flow and recirculation flow were measured daily. The detention time in the aerator was calculated on the basis of the influent flow divided into the aeration tank volume (33 liters), with no regard to the recirculated flow. The M.L.S.S., Effl. S.S., and SVI were determined in accordance with Standard Methods (Reference 13) from samples obtained at the same time as the grease samples.

The results of the grease analyses are reported in Table V-II. Grease addition started on Day 2, with the first set of analyses being run on Day 3. The accumulated values for the feed are based upon the feed grease determination and the flow rate except when pumping difficulties were encountered. During those periods the accumulated values were based upon the volume of concentrated feed consumed during the testing interval. The mixed liquor value under grease "accumulated values" in Table V-II is the total weight of grease present in the aeration tank at the time of sampling (M.L. grease concentration aeration tank volume). The amount of grease in the sedimentation tank is determined by multiplying the effluent grease concentration by the sedimentation tank volume. Since the grease concentration in the sedimentation tank is nonhomogeneous (because of sediment at the base, more clarified liquor in the center, and floating material on the surface), this assumption is felt to be on the conservative side. Effluent accumulated values are based upon effluent concentrations and effluent flow rates.

C.O.D. concentrations of the feed were measured daily and were found to be between 275-300 mg/l. Effluent C.O.D. analyses were made, and they were found to be proportional to the effluent suspended solids and in the range of 75-200 mg/l. Feed pH was 7.6 - 7.8, M.L. pH 6.9 - 7.0, and effluent pH 6.7 - 7.0 throughout the experiment. Mixed liquor temperature was a constant 21° C.

Since the purpose of this experiment was to determine the grease removal properties for short aeration times, the results will be discussed from the grease results, with the other data used as supporting material. Days 3 and 4 saw good grease removal when influent and effluent grease concentrations are compared, after which the removal became very poor. This grease accumulated in the aeration tank, which is also reflected in the increased M.L.S.S. concentrations during this period. Examination of the solids confirmed the fact that a large part of the solids consisted in grease. A steady accumulation of grease took place in the aeration tank until Day 7, after which the amount decreased. By visual observation it appeared that the grease accumulated until a limiting concentration was reached in the aeration tank, after which the grease "sloughed" from the tank and became enmeshed with the activated sludge. The resulting mass was "bulky" and over a period of a few days was discharged from the aeration tank. One day (Day 8) had more grease in the effluent than in the feed, indicating that the phenomenon described above is probably operating.

After the sloughing period, removals in the plant improved, probably due to a reaccumulation process taking place. However, by this time the vast majority of the activated floc was lost, and rebuilding of such a floc was very difficult under the short aeration period being studied (about 1.3 hours).

The results of the experiment, as reported in Table V-II, showed that 83 percent of the added grease left the treatment unit with the effluent and that an additional 13,5 percent of the grease accumulated in the aeration and sedimentation tanks during the nine-day period. This accounts for a total of 96.5 percent of the added grease remaining unconverted.

On the basis of these results it can be concluded that little microbial metabolism of the grease took place during the nine-day experiment.

However, a series of separate jar tests was run in order to determine the time requirement to render the floatable material settleable. The results of these tests showed that a minimum of three weeks of constant aeration was required in order to settle out all the material, with no feed being introduced to the units for the entire length of time. Invariably, the pH of the mixture dropped to about 5.5 during the first few days of the test, after which is rose to about 7.0 to 8.0, where it remained throughout the remainder of the tests. A treatment of this nature was thought to be too expensive to be considered on a plant scale. In addition, a large amount of grease continued to remain in suspension and, when diluted, became de-emulsified and rose to the surface causing slicks.

Conclusions

1. Activated sludge treatment for the removal of grease under short aeration times is considered to be an ineffective treatment method.
2. The applied grease was not metabolically degraded to any significant extent.
3. The process can remove some grease, but the mechanism is thought to be a combination of adsorption on surfaces and air-flotation.
4. Flotation and removal of the floatables may be a significant treatment method.
5. When sloughing of the accumulated mat of sludge and grease takes place, the ratio of grease to sludge is often such that the combined mat will float.

PHYSICAL METHODS FOR FLOATABLES REMOVAL

At conventional waste treatment plants, the flotation and collection of floatable material is achieved by a sedimentation basin. However, short circuiting, density current and other hydraulic disturbance result in inefficient removal of floatables. Also, slow rising particles with marginal velocity are carried out into effluent.

The flotation process can be improved by increasing the detention time or by increasing the rising velocity of the floatable matter. The rising velocity can be increased by attaching air bubbles to the floatable particles (aeration) by increasing the density difference between floatable matter and suspending medium, such as sea water dilution or by increasing the particle size by coagulation. Of these possibilities it at first appeared that either prolonged flotation times or fine bubble aeration, or the creation of larger floatable particles by coagulation, or de-emulsification by sea water addition*) would be the most promising.

The Effect of Aeration, Sea Water Dilution, and Flotation Time on Floatables Removal

An experiment was therefore designed to test the influence of aeration, sea water dilution, and flotation time upon the separation of floatable material and grease from raw domestic sewage prior to its discharge into the marine environment. The various treatments studied are outlined in Table V-III below:

TABLE V-III

PHYSICAL TREATMENT METHODS STUDIED FOR MARINE SEWAGE DISPOSAL

Method	Units	Levels
Aeration	Time, min.	0, 5, 25
Sea Water Dilution	Percent	0, 15, 30
Flotation	Time, min	5, 30

The experiment was performed in the manner already described in Chapter IV, which tested all eighteen of the above possible combinations, with four replications of each combination.

The statistical design used in this experiment was arranged in blocks where six individual tests were performed at one time on a single source of sewage. The fact that different sewages were used in the different blocks did not affect the outcome of the experimental statistics, even though the averages in each block were often quite different

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*)The decision to investigate the effect of sea water as a treatment method was based upon the first set of experiments described in Chapter IV showing that mixing of sea water and a waste with low concentration of floatable material resulted in a significant increase in the amount of 0.7 mg/l or 39 percent of total.

The second set of experiments described in the previous chapter investigating the effect of sea water on a waste containing a high concentration of floatable material was made after the experiment described in this chapter had been completed.

Apparatus

Six Flotation Funnels as described in Chapter IV were used in this experiment, the funnels being provided with a fritted glass diffuser disc. The six reactors used for this experiment were originally designed for testing the floatable material in sewage or industrial wastes. A brief description of these reactors seems appropriate in this report.

The fritted glass diffuser discs were fitted to each flask so that the maximum "dead space" under the diffuser was approximately 75 ml. The porosity used on these diffusers was M Porosity (nominal pore size of 10-15 microns).

The flasks and paddles were Teflon coated to prevent adherence of grease to their surfaces. The fritted glass discs, and their stems, were not coated but any grease adhering to these surfaces was found to be negligible.

The analytical tests used in this experiment were the n-hexane liquid-liquid grease extraction procedure and the floatable procedure described in Appendix A and Appendix B.

Experimental Procedure

Raw sewage used in this experiment was obtained from the Richmond-Sunset Sewage Treatment Plant in San Francisco. This plant serves an area of the city which is predominantly domestic and light commercial, with a very small industrial waste flow. The sewerage systems contributing to this plant are combined sewers, but only a negligible amount of precipitation fell throughout the duration of our sampling program. Enough raw sewage was collected at one time to allow two blocks of experiments to be run (a total of 12 separate tests). The usual procedure was to collect the sewage late in the day and to run one block of tests the following morning, and the second block on the morning after. Therefore, half of the tests were run on sewage about 16 hours old, and the other half on sewage approximately 40 hours old.

Each experiment and the accompanying analytical testing took nearly a complete working day. The first step was to transfer sufficient sewage to a large carboy where it was subjected to complete mixing for a period of 15 minutes. During this period, salt water was added to those flasks called for in the experimental design.

The next step was to fill each reactor flask to the three liter mark with the mixed raw sewage. The mixing flask was fitted with a draw-off stem located approximately three inches from the base of the flask. The reactor flasks were filled in a random order to prevent any differences between the times and order of filling from introducing a bias in the statistical analysis of the final results.

Immediately after all six flasks were filled, the paddles were introduced into the flasks and the contents were subjected to a five-minute stirring at 30 RPM. The purpose of this step was to be certain that the salt water became completely mixed with the sewage.

The next step in the procedure consisted of placing the gas diffusers into those reactors called for in the experiment. In order to be reasonably certain that each diffuser was under identical conditions of pressure and air flow (0.07 cfm/gal), a large manifold was built with six identical exit orifices to which six diffusion discs were attached with equal lengths of flexible tubing. Thus the compressed air applied to each reactor was identical. The manifold was equipped with a pressure gauge located in the center and a flow meter on the influent air line between a pressure regulator valve and the manifold.

Since only four reactors were subjected to aeration during any single experiment, and two of these for only part of the experiment, a large vessel containing water was provided adjacent to the experimental apparatus into which the diffusers not being used could be placed so that all six discs would have the same hydrostatic pressure to work against. Although the probability of all the diffusers being identical was very remote, the experimental design was such that variations between reactors were taken care of by randomization of the various replications among reactors.

Immediately prior to the flotation phase (either after the initial mixing for tests with no aeration or after aeration for the remainder of the tests), the stirrers and diffusers were removed and the sewage was allowed to settle for a predetermined period (5 or 30 minutes). At the end of this flotation time, 1.8 liters were withdrawn from the bottom of the reactor and wasted. The next liter was transferred to a grease determination sample-reaction bottle, and the remainder of the manipulations were carried out for the determination of floatable material described in Appendix B.

Results

The results of this experiment are tabulated in Tables V-IV, V-V, and V-VI. Table V-IV gives the results of grease concentrations in mg/l of the middle liter of material in the reactor, and Table V-V gives the results of the floatable material in mg/sample, or mg/3 liters. Where experiments were run with the addition of sea water, the results shown have been corrected for this dilution.

Tables V-IV and V-V are arranged so that all information about the experiment is given. Roman numerals I through IV represent the four replications. The small letters a, b, and c represent the blocks within replications. The numbers in parenthesis are the code for the experiment. The first number is the level for aeration, the second number is the level for sea water, and the last is the level for flotation time. Referring to Table V-III for the various levels, (210) represents 25 min. aeration, 15 percent sea water, and 5 min. flotation time. Likewise (011) represents 0 aeration, 15 percent sea water, and 30 min. flotation time. The remainder of the numbers are the results of the experiment in units already discussed.

The above results were subjected to the analysis of variance described in detail by Cochran and Cox in "Experimental Design" (Reference 11). Table V-VI shows the results of this analysis.

TABLE V-IV

GREASE RECOVERY
mg/l grease in middle layer

REPLICATE I

Ia	Ib	Ic
(100) 68.2	(200) 56.3	(000) 80.9
(210) 59.4	(010) 52.9	(110) 76.0
(020) 61.9	(120) 73.1	(220) 64.0
(201) 64.1	(001) 54.2	(101) 79.9
(011) 32.6	(111) 44.9	(211) 35.1
(121) 47.6	(221) 52.5	(021) 87.3
333.8	333.9	423.2

REPLICATE II

IIa	IIb	IIc
(200) 45.6	(000) 56.3	(100) 51.1
(010) 79.8	(110) 40.2	(210) 37.3
(120) 59.9	(220) 80.5	(020) 36.1
(101) 58.7	(201) 36.5	(001) 29.6
(211) 77.2	(011) 53.2	(111) 36.2
(021) 83.3	(121) 39.2	(221) 32.0
404.5	305.9	222.3

REPLICATE III

IIIa	IIIb	IIIc
(100) 28.6	(200) 56.9	(000) 40.3
(010) 43.6	(110) 37.0	(210) 61.5
(220) 29.9	(020) 61.2	(120) 71.6
(201) 37.6	(001) 48.3	(101) 52.5
(111) 21.0	(211) 63.1	(011) 63.1
(021) 31.3	(121) 33.2	(221) 53.5
192.0	299.7	342.5

REPLICATE IV

IVa	IVb	IVc
(200) 55.2	(000) 48.7	(100) 51.4
(110) 43.6	(210) 46.8	(010) 27.8
(020) 51.4	(120) 40.8	(220) 41.7
(101) 34.9	(201) 18.0	(001) 21.6
(011) 29.2	(111) 23.2	(211) 32.4
(221) 22.6	(021) 51.5	(121) 44.1
236.9	229.0	219.0

Σ I = 1090.9

Σ II = 937.2

Σ III = 834.2

Σ IV = 684.9

Grand Σ = 3542.7

TABLE V-V

FLOATABLE MATERIAL RECOVERY
mg of floatables per 3 liters of sample

REPLICATE I

Ia		Ib		Ic	
(100)	53.1	(200)	67.1	(000)	33.8
(210)	34.8	(010)	92.7	(110)	54.1
(020)	37.9	(120)	76.1	(220)	61.4
(201)	23.5	(001)	110.1	(101)	31.4
(011)	49.5	(111)	106.9	(211)	62.6
(121)	35.2	(221)	167.2	(021)	59.7
	234.0		620.1		303.0

REPLICATE II

IIa		IIb		IIc	
(200)	49.1	(000)	34.9	(100)	12.9
(010)	29.5	(110)	43.8	(210)	15.4
(120)	35.5	(220)	39.1	(020)	22.0
(101)	30.7	(201)	40.6	(001)	83.2
(211)	41.6	(011)	49.0	(111)	14.9
(021)	49.1	(121)	56.2	(221)	10.5
	235.5		263.6		158.9

REPLICATE III

IIIa		IIIb		IIIc	
(100)	6.9	(200)	112.9	(000)	49.4
(010)	11.0	(110)	114.0	(210)	49.8
(220)	7.4	(020)	135.8	(120)	33.7
(201)	13.3	(001)	95.2	(101)	51.3
(111)	9.4	(211)	90.6	(011)	61.5
(021)	12.3	(121)	91.9	(221)	55.1
	60.3		640.4		300.8

REPLICATE IV

IVa		IVb		IVc	
(200)	12.7	(000)	14.0	(100)	8.9
(110)	15.9	(210)	13.5	(010)	12.4
(020)	21.6	(120)	17.8	(220)	9.7
(101)	17.0	(201)	21.4	(001)	13.9
(011)	22.1	(111)	16.5	(211)	10.9
(221)	13.6	(021)	13.2	(121)	14.1
	102.9		96.4		69.9

Σ I = 1157.1
Σ II = 658.0
Σ III = 1001.5
Σ IV = 269.2
Grand Σ = 3085.8

TABLE V-VI

ANALYSIS OF VARIANCE FOR GREASE AND FLOATABLE REMOVAL

Source	Degrees of Freedom	Floatables S.S.	Floatables M.S.	Grease S.S.	Grease M.S.
Replications	3	25,932	8,644	5,317	1,772
Blocks within replications	8	43,537	5,442	5,224	653
Aeration (A)	2	573	287	129	65
Sea Water	2	167	84	371	186
Flotation	1	582	582	1,187	1,187**
Aer. & Sea Water	4	682	171	827	208
Aer. & Flotation	2	311	155	113	57
Sea Water & Flotation	2	23	11	2	1
Aer., Sea Water & Flotation	4	3,156	789*	595	149
Error	43	9,152	212	6,782	158
	71	84,124		20,547	

*Significant at 5% level.

**Significant at 1% level.

Above values determined by the "F" test.

The above results show that in terms of the quantity of floatable material recovered, the only factor found to be significant was the interaction of the three factors investigated. However, of all other factors, flotation alone came quite close to being significant (as seen by the magnitude of the mean square for this factor).

The results of the grease removal showed flotation time to be significant at the 1% level, with no other factors approaching significance.

It should be added that significance was found in both replications and blocks within replications, a result which reflects the variations in sewage from day to day.

Discussion

The results show that of the methods tested only flotation showed any significant effect on the removal of grease and on the removal of floatable material. This result is not unexpected, as it is quite logical that more particles will rise to the surface in proportion to the period of quiescence. Such a reaction is simply in accordance with Stokes' Law.

The purpose of sea water addition was to test the hypothesis that this material was a good de-emulsifying agent because of the presence of multivalent ions. However, the tests being reported in this paper did not confirm this fact. A possible explanation for this result might well be seen when the number of multivalent ions in sewage are considered. The presence of these ions has probably de-emulsified all the grease possible by the time the sewage has reached the treatment plant, and the addition of more multivalent ions would be redundant as a treatment possibility.

The factor of increasing the density of the solution by the addition of sea water is nearly negligible when it is considered that pure sea water has a density of 1.025. Thus a dilution with 15 or 30 percent sea water will increase the density of the mixture less than one percent compared to the sewage.

The fact that fine bubble aeration had no significance was an unexpected result. However, studies by others have shown that preaeration is of little value in the removal of grease and floatable material. Thus, the results of this experiment seem to substantiate the results found by Seidel and Baumann, (Reference 14).

As an added dividend from this experiment, two observations were made that might have some significance. The first is concerned with the effect of holding the sewage prior to running the experiment. Table V-VII is a summary of the dates the sewage was collected, the dates it was used for the experiment, and the average values found for grease and floatables on those analyses.

TABLE V-VII

SEWAGE COLLECTION DATES AND AVERAGE FLOATABLES AND GREASE FOR LOTS

Date Collected	Test Date	Average Grease	Average Floatables
23 Nov	24 Nov	70.5	50.5
23 "	25 "	67.4	39.3
30 "	1 Dec	37.5	26.5
30 "	2 "	32.0	10.5
2 Dec	3 Dec	49.9	106.7
2 "	4 "	57.1	50.1
7 "	8 "	39.5	17.2
7 "	9 "	38.2	16.1

Table V-VII shows that, with one exception, the average grease contents of the lot was lower on the older sewage than on the relatively fresh sewage. Also, the amount of floatable material was, in all cases, lower on the older sewage than on the fresh. Although these results are only estimates with little statistical validity, they do seem to present a trend. Thus, from these results it might be concluded that grease decreases, as does the total floatable fraction. This result appears to be in contradiction to itself because if the grease becomes less, it would seem that the floatables would increase by a proportional amount, which does not appear to be occurring.

When examining the results presented, it is quite apparent that the floatable content of the sewage is much more variable than the grease content, even considering that some treatment is being effected. The average value of all grease determinations was found to be 49.2 mg/l with a standard deviation of 16.9, while the average floatable concentration was 42.9 mg/3 liters with a standard deviation of 34.2. The only conclusion that is able to be reached with these results is simply that the quantity of floatable material is not nearly as consistent in domestic sewage as the grease content of the sewage. The significance of the above is doubtful at this time.

Conclusions

The following conclusions may be drawn from this experimental study:

1. Preaeration of domestic sewage has no significant effect on the removal of grease or floatable material.

2. Addition of up to 30 percent sea water is not a significant method for removal of floatable material. (Although for some types of domestic waste, the addition of very large amounts of sea water produced significant effect.)

3. Flotation time is the most significant factor in the removal of grease and floatable material.

4. None of the treatment methods studied appear to have any advantage over the presently common method of plain sedimentation.

PRESSURE FLOTATION

The attempts to increase the rising velocity of floatable matter by fine bubble aeration or by sea water dilution were not successful. According to the study by Seidel and Baumann (Reference 14) the ineffectiveness of preaeration in the removal of grease particles may be due to the relatively large size of the air bubbles compared to the grease particles to be removed. If the size of the air bubbles used in the flotation process is the determining factor, the removal of grease (and other hydrophobic particles) might be facilitated by the introduction of microscopic air bubbles. The generation of small air bubbles can be achieved either by mechanical shear of a propeller or by precipitation of gas bubbles from a solution supersaturated with the gas. Vrablik (Reference 15) described the former method as dispersed-air flotation and the latter as dissolved-air flotation. The bubbles produced by dissolved flotation is generally smaller than 80 micron in diameter, while bubbles by dispersed-air flotation reach up to 1000 micron.

The theory of flotation was developed from kinetic consideration of gas bubbles and suspended solids by Gaudin (Reference 16) and Klassen and Mokrousov (Reference 17). Flotation process has been widely applied to the collection of minerals from ores using dispersed flotation process. The present status of this process in the handling of minerals was described by Fuerstenau et al (Reference 18).

However, in the field of sewage and industrial waste treatment, the dissolved-air flotation rather than the dispersed-air flotation was used successfully. Depending on the means of producing a pressure differential for the generation of tiny gas bubbles, the dissolved-air flotation can be divided into pressure flotation and vacuum flotation. The study of the pressure flotation process on refinery waste waters by Rohlich (Reference 19) showed that the process removes 50-78% of oil in the waste water without the use of other chemicals. Garrison and Geppert (Reference 20) report 93% removal of grease on packinghouse waste by pressure flotation. Also, vacuum flotation was used by Logan (Reference 22) and Mays (Reference 23) on industrial wastes.

However, the effluent from the dissolved-air flotation process on industrial waste has generally more grease than the untreated, domestic wastes from which this project intends to remove floatables. The effectiveness of the dissolved-air flotation on the removal of floatables has not been investigated so far.

It was therefore decided to conduct a preliminary laboratory investigation of the pressure flotation process on both raw and settled sewage.

Pressure Flotation of Raw Sewage

The purpose of this part of the investigation was to collect preliminary data to be used for the planning of the pilot plant experiments to be conducted during the third project year.

A pressure aeration unit was constructed from an 8-liter surplus oxygen tank. The tank was provided with an air inlet, an air release valve, and a pressure gauge as shown in Figures V-4 and V-5.

Experimental Procedure: A 7-liter sample of sewage was placed in the tank and saturated with air at 38 psi (by bubbling compressed air through the sample for 10 minutes). Then, the sewage was released into one of the Flotation Funnels (described in Chapter IV) through the bottom 3-way valve. When the sewage entered the Flotation Funnel, the pressure was released and fine air bubbles were formed. The sewage was allowed to settle (and float) in the Flotation Funnel for 30 minutes, after which the first liter was wasted from the bottom of the Funnel. The next 1.8 liters were collected and saved for the analyses of suspended solids and suspended grease. The remainder portion (about 200 ml) of the liquid underneath the layer of floated material was then wasted. The floated matter in the form of a foam was washed down through the bottom of the funnel, collected on a preweighed filter paper, dried, and weighed. The mixing step and the washing step in the floatables test were omitted in this particular application because any disturbance of the foam might break up the emulsion and settle a portion of the floated matter.

A second sample from the same batch of sewage was treated in exactly the same manner with the exception that the air saturation at the elevated pressure was omitted. The efficiency of the pressure flotation was then determined as the difference in floated material between the pressurized and the non-pressurized sample.

Results: Three different types of raw sewage were obtained from plants in the San Francisco Bay Area and tested according to the procedure outlined above. The results of the tests are shown in Table V-VIII. All three

PRESSURIZING UNIT FOR DISSOLVED-AIR FLOTATION PROCESS

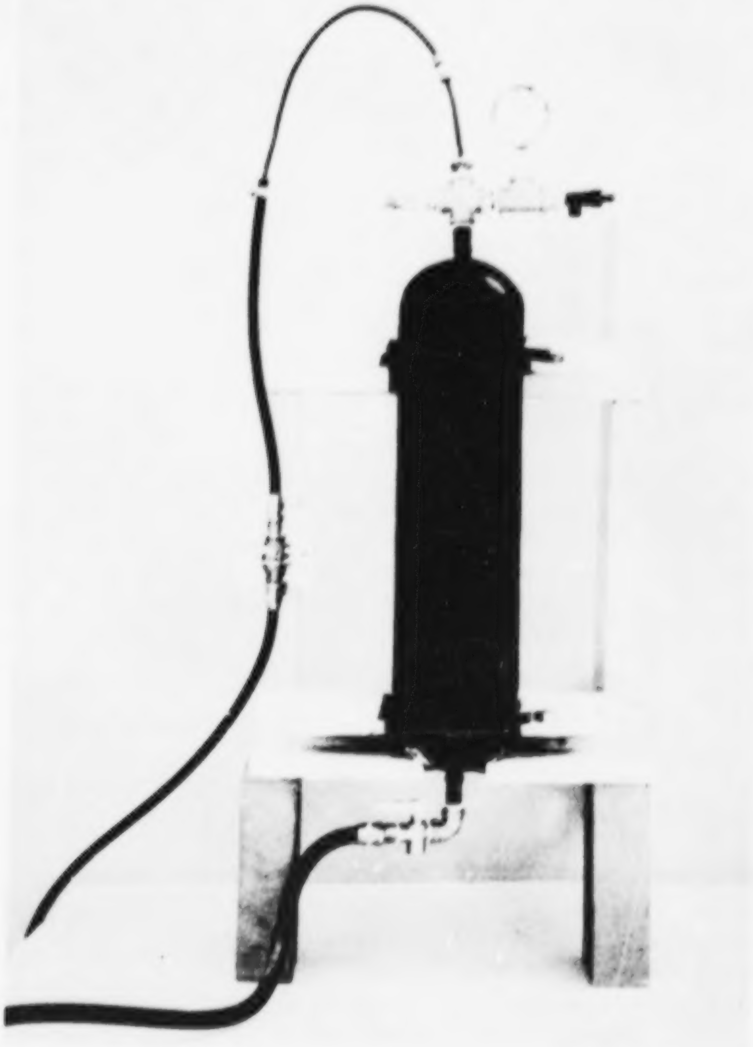


FIGURE V-5

FLOW DIAGRAM OF PRESSURE FLOTATION UNIT

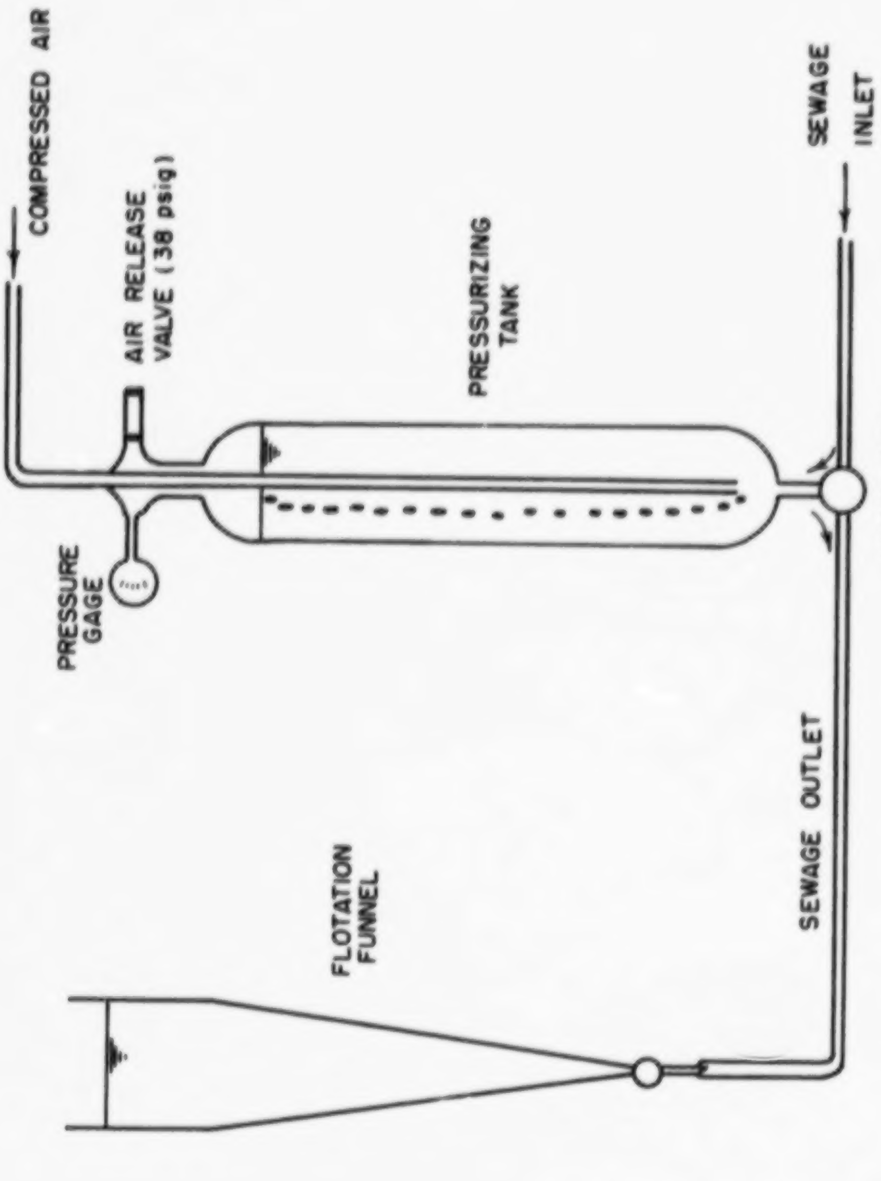

COMPRESSED AIR
AIR RELEASE VALVE (38 psig)
PRESSURE GAGE
PRESSURIZING TANK
SEWAGE INLET
THREE WAY VALVE
SEWAGE OUTLET
FLOTATION FUNNEL

TABLE V-VIII

THE EFFECT OF PRESSURE FLOTATION COMPARED TO GRAVITY FLOTATION ON THREE DIFFERENT RAW SEWAGES

SOURCE OF SEWAGE		Plant #1		Plant #2		Plant #3	
TREATMENT		Pressure Flotation	Gravity Flotation	Pressure Flotation	Gravity Flotation	Pressure Flotation	Gravity Flotation
Floated Matter	mg/l	115.2	9.1	141.8	2.2	125.0	2.2
Suspended Solids*)	mg/l	122.	139.	229.	158.	130.	70.
Suspended Grease	mg/l	59.8	72.9	35.7	34.7	35.9	32.4
Settleable Solids	ml/l	< 0.1	~ 10	< 0.1	~ 10	< 0.1	~ 10

*) Suspended Solids are solids which stayed in suspension after 30 minutes of quiescent settling.

samples showed a large increase (10-15 times) in the amount of floated material collected by pressure aeration as compared to the plain gravity flotation process. The amounts of suspended solids or suspended grease were found to be increased in two of three cases when pressure flotation was compared with conventional gravity flotation. The amount of settleable solids decreased in pressure floated samples, and an amount equivalent to the settleable material was floated by this process. This was also shown by the fact that the major part of the floated material could be reconverted into sinkables by water spray or mechanical stirring.

The flotation and collection of settleable suspended solids together with the true floatable matter increases the amount of scum to be disposed of. Despite this apparent disadvantage, this method suggests an interesting new approach to the handling of floatables and sludge. The collection of sludge and floatables in one unit by pressure flotation may give the following advantages:

1) The detention time can be shortened and the space requirement will be reduced.

2) The scum and sludges obtained are, in many cases, thicker than the same by a sedimentation basin.

3) The removal efficiency of floatables may be greater with the rising mat of sludge than without it.

The disadvantages anticipated are:

1) The flotation unit usually does not give effective removal of settleable suspended solids compared to a gravity settling tank. This may result in an increased concentration of suspended solids in the effluent compared to that from a sedimentation basin.

2) If the collected scum (floatables and sludge) is to be digested before the disposal into the marine environment, the increased scum layer in the digester due to the efficient collection of floatables may pose a problem. Also, the digested sludge may still contain a greater amount of grease than before which can cause more floatables in the ocean.

3) If land disposal of the collected scum is contemplated, the volume to be disposed is considerably greater than the floatable material alone.

A careful evaluation of the above factors based on pilot plant scale investigations will be necessary.

During these tests is was observed that the floatables layer formed by the pressure flotation process was much more stable than the non-pressurized floatables layer. The lack of stability of floatables in sedimentation tanks is believed to be a very significant factor in their inefficient performance; (from field observations described in Chapter VI). In order to decrease the amount of floated matter but still obtain a stable scum layer, pressure flotation of settled sewage was investigated.

Pressure Flotation of Settled Sewage

Samples of raw sewage were collected from the same three sources, allowed to settle one hour in the laboratory, after which the supernatent was transferred to the pressure cylinder and aerated as already described.

The total amount of floated material was determined for both the pressure aerated samples and the non-aerated controls. In addition, the amount of grease in the floated matter was determined. Suspended solids and suspended grease determinations were not carried out, since the change in floated material corresponds to the change in suspended material when settleable material is absent.

Results: The results of the floatables determination are reported in Table V-IX and show a three-fold increase in floated matter by pressure flotation as compared to ordinary gravity flotation. The percent grease in the floated material varied between 50 and 6 percent for pressure floated material, and between 48 and 9 percent for plain floated material, for the three different types of sewage. The limited results do not indicate any increased removal of grease by pressure flotation relative to the removal of non-grease suspended material.

Conclusions to the Pressure Flotation Experiments

1. Pressure flotation of raw sewage causes otherwise settleable suspended solids to rise to the surface. The amount of scum collected by this method may be up to ten times larger than the amount collected without pressure flotation. The practicality of this method on raw sludge for the sake of floatables removal needs a careful evaluation based on a pilot plant scale study.

2. Pressure flotation of settled sewage appears promising for floatables removal because of the formation of a stable foam layer with only a limited increase in solids to be disposed on land. Such a stable foam layer can possible be more completely removed than the layer obtained by plain sedimentation.

SUMMARY AND CONCLUSIONS TO CHAPTER V

1. Prolonged flotation times will enhance the removal of floatable material whereas aeration and the addition of up to 30 percent sea water appear to be without effect.

2. Based on laboratory tests, pressure aeration appears to increase the amount of floated material by as much as ten times for raw sewage and about three times for settled sewage compared to gravity flotation based upon laboratory results.

3. The laboratory data indicate that promising treatment methods for marine disposal are pressure flotation of raw sewage, improved gravity retention focused on improving floatable solids removal, and gravity retention followed by pressure flotation of the effluent.

TABLE V-IX

THE EFFECT OF PRESSURE FLOTATION COMPARED TO GRAVITY FLOTATION ON SETTLED SEWAGE

The results reported in the table are obtained on duplicate samples from each of the plants; one of the samples treated by pressure flotation and the other by gravity flotation.

SOURCE OF SEWAGE		Plant #1		Plant #2		Plant #3	
TREATMENT		Pressure Flotation	Gravity Flotation	Pressure Flotation	Gravity Flotation	Pressure Flotation	Gravity Flotation
Floated Matter	mg/l	13.3	4.4	28.3	8.6	18.9	4.6
Grease in Floated Matter	mg/l	6.7	2.1	5.2	1.9	1.2	0.4
Percent Grease in Floated Matter		50%	48%	18%	22%	6%	9%
Settleable Solids	ml/l	nil	nil	nil	nil	nil	nil

VI. CONCENTRATION OF FLOATABLE MATERIAL IN DOMESTIC SEWAGE AND EFFICIENCY OF EXISTING REMOVAL METHODS

INTRODUCTION

The bench scale laboratory tests described in Chapter V showed plain gravity flotation and pressure/vacuum flotation to be the two most promising processes for the removal of floatable material from domestic waste waters before disposal into the marine environment. A survey of the literature subsequently showed that little information existed regarding the efficiency of removal of floatable material on a plant scale. Furthermore, no information was found concerning the range of concentration of floatable material in domestic sewage.

A study was therefore conducted at two existing treatment plants in the San Francisco Bay Area with the dual objectives of determining the level of floatables concentration in raw sewage and the efficiency of their floatables removal methods. This will serve as background information for the final evaluations to be completed during the third year of study.

The first plant studied, the Richmond Water Pollution Control Plant (Richmond WPCP) employed preaeration followed by gravity flotation for the removal of floatable material. The second plant, the South San Francisco-San Bruno Water Pollution Control Plant, employed a vacuum flotation process for floatables removal.

In addition, a program of floatables determination was undertaken with the Los Angeles County Sanitation Districts and with the City of Los Angeles.

PREAERATION AND GRAVITY FLOTATION

Plant Description

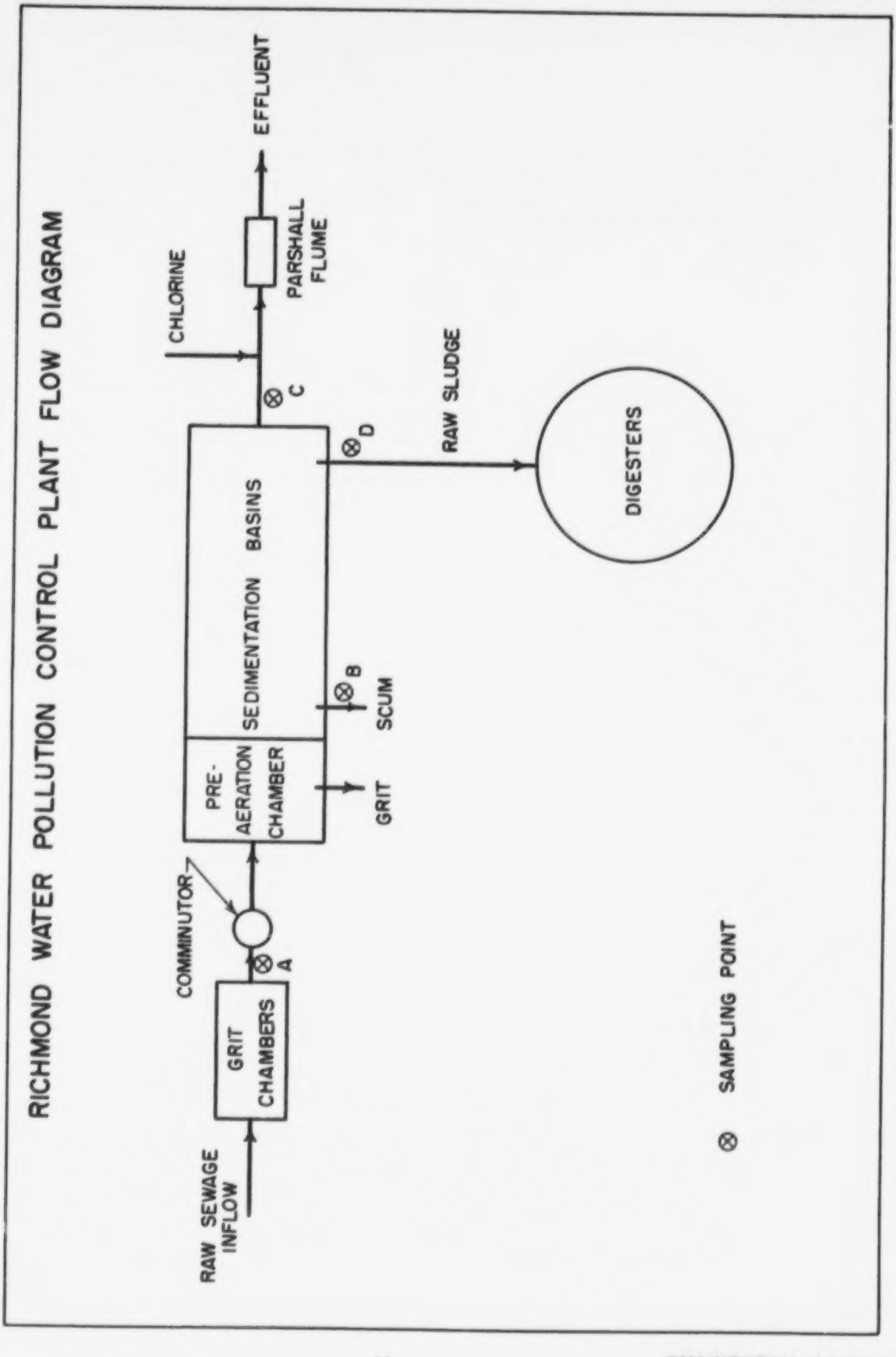
The Richmond Water Pollution Control Plant is a primary treatment plant with an average design flow of 12.2 MGD. The flow diagram for the plant is shown in Figure VI-I; and the design characteristics pertinent to the floatables removal system are presented in Table VI-I. The plant employs a water jet system for the collection of floated material near the inlet of the settling tank, where the material is subsequently transferred to a special truck and buried on land. This system permitted the attempt to obtain a mass balance for the floatable material in the plant as described in Table VI-I. The sewage is mainly domestic except during the later summer when the plant receives considerable amounts of cannery waste. The sampling reported here was conducted before the canning season.

Sampling Program

With the objective of obtaining information about the concentration of floatable material in both raw sewage and primary effluent together with information about the efficiency of the floatables removal process, the following sampling program was selected:

A sample of influent, effluent, and raw sludge was collected every two hours during two 24-hour periods, and the amount of floatable material in the

FIGURE VI-1



RICHMOND WATER POLLUTION CONTROL PLANT FLOW DIAGRAM

RAW SEWAGE INFLOW
GRIT CHAMBERS
COMMINUTOR
A
PRE-AERATION CHAMBER
GRIT
SEDIMENTATION BASINS
B
SCUM
D
RAW SLUDGE
DIGESTERS
C
CHLORINE
PARSHALL FLUME
EFFLUENT
⊗ SAMPLING POINT

TABLE VI-I

PERTINENT DESIGN CHARACTERISTICS FOR RICHMOND WATER POLLUTION CONTROL PLANT

Average design flow	12.2 MGD
Prechlorination rate	5 ppm
Comminuters, clear openings	3/8 inch
Preaeration tanks	
Number	2
Width	36 feet
Length	45 feet
Average water depth	11 feet
Air supplied	500 cu ft/min
Sedimentation tanks	
Number	2
Width	36 feet
Length	164 feet
Average water depth	11 feet

samples determined immediately. Composite samples of the three streams were also collected for the determination of hexane extractable material. In addition to the composite influent sample, separate samples of the influent were collected every second hour during the first 24-hour period and analyzed for hexane extractable material to obtain information about the daily variation in grease concentration during a 24-hour period. The influent, effluent, and sludge sampling points are marked A, B, and C, respectively, on Figure VI-1.

Since the collection of a representative sample is one of the major concerns in floatables determination programs, it was necessary to collect the influent sample before comminution and the preaeration basin; the actual plant layout did not permit the collection of a representative sample between the preaeration basin and the flotation basin. Since the laboratory tests had shown (Chapter V) that conventional preaeration had no significant effect on the removal of floatable material, this sampling location was believed adequate.

Sampling and Analytical Methods

To insure the collection of representative samples of floatable material the following sampling procedure was used: A 2½ gallon steel bucket (paint can) was provided with a bottom outlet consisting in a four inch long piece of one inch diameter pipe. The pipe was cut off in a 45° angle at one end and welded to the bucket as shown in Figure VI-2.

This bucket was used to collect the samples at points where there was a complete mixing of the individual flows. The buckets were then carried to the laboratory and their content mixed by means of a laboratory propeller mixer in such a way that the floatables were uniformly distributed throughout the whole sample. The propeller shaft was inclined slightly from the vertical position to avoid the development of a vortex. A three liter sample was drawn off from the bucket directly into a Flotation Funnel and the concentration of floatable material determined as described in Appendix B. In addition to the floatables sample, a sample proportional to the flow was composited during each 24-hour period of hexane extractable material. During the first 24-hour period, individual samples of the influent were also collected every second hour and analyzed individually to determine the variation in the concentration of hexane extractable material over a 24-hour period.

Four times during the first 24-hour period and once during the last 24-hour period small samples of the floatable material were collected in preweighed trays and the percent water determined by drying in a 40° C oven to constant weight. This determination was followed by the determination of the hexane extractable material in the dried sample.

Concurrently with the sampling, the effluent flow was determined by the effluent flow meter, and the flow of raw sludge estimated from the sludge pump schedule combined with the known rate of pumping. No provisions existed for measuring the influent flow, for which reason the measured effluent flow two hours later was used in the calculations of the total amount of floatable material entering the plant.

Sampling Results

The overall results of the sampling are shown in Figure VI-3 and in Tables VI-II and VI-III; the detailed results are presented in Appendix D. The average concentration of floatable material in the influent was 5.2 mg/l and 4.9 mg/l, respectively, with corresponding average concentrations of 2.9 mg/l and 2.2 mg/l, respectively, in the effluent during the two 24-hour periods. These results will be discussed further in relation to the results obtained from the vacuum flotation process.

The comparison between composite samples for determination of hexane extractable material and the analysis of the individual samples is presented in Table VI-IV for the first 24-hour period. The total amount of hexane extractable material in the influent was 4,336 lbs based upon the individual samples and 4,623 lbs based upon the composite sample. It was concluded from this result in combination with the result obtained in Chapter III that composite samples would be adequate for the future treatment plant surveys to be conducted during this study.

In addition, an attempt was made to obtain a mass balance for floatable material and hexane extractable material during the two periods. Although these materials balances are prepared using the existing plant flow meters, etc., the accuracy of which was not possible to verify during the available time, these are, nevertheless, two observations worthwhile mentioning.

FIGURE VI-2

FLOATABLES SAMPLER WITH MIXER

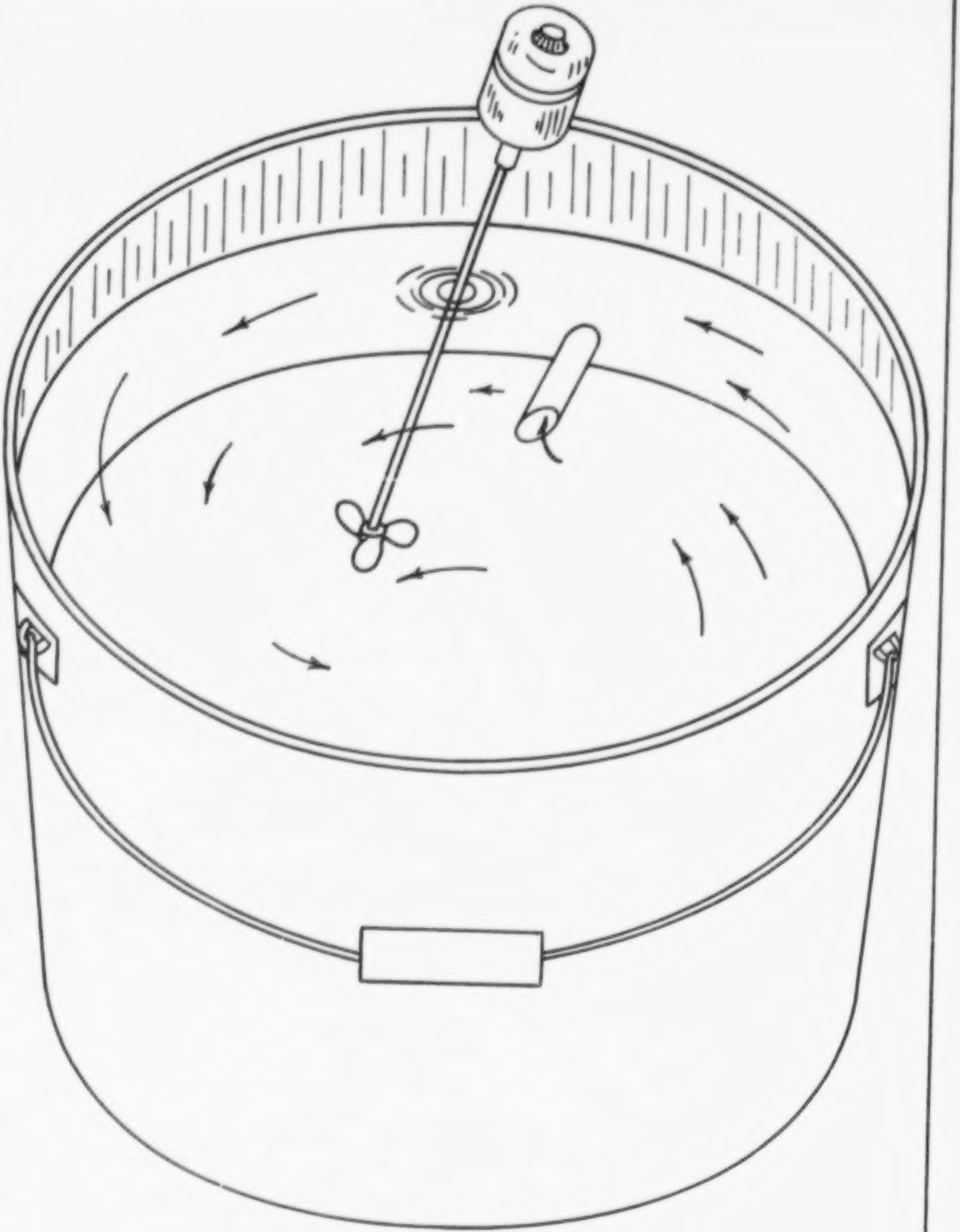
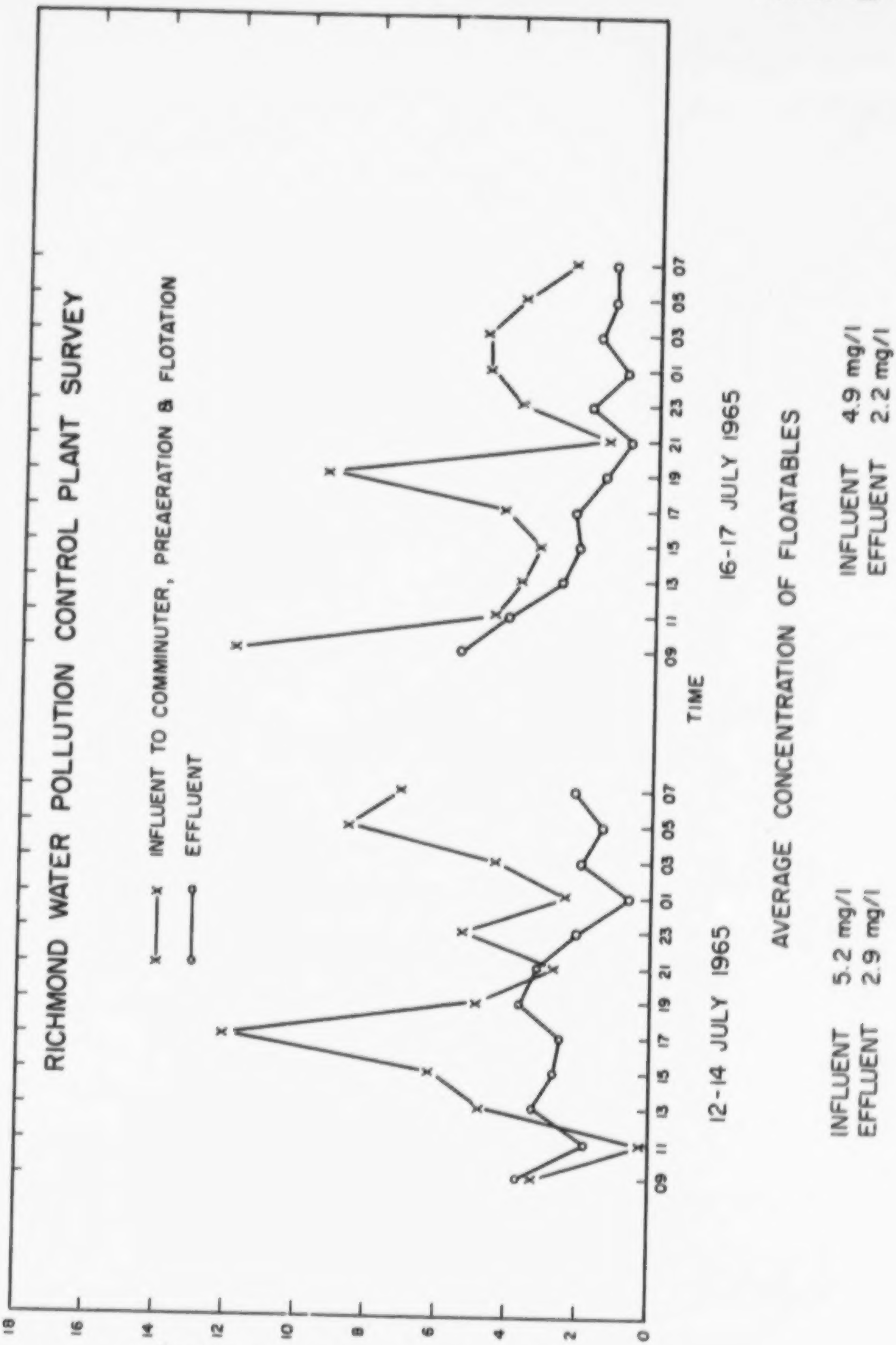


FIGURE VI-3


RICHMOND WATER POLLUTION CONTROL PLANT SURVEY
x—x INFLUENT TO COMMINUTER, PREAERATION & FLOTATION
o—o EFFLUENT
FLOATABLES CONCENTRATION, mg/l
0
2
4
6
8
10
12
14
16
18
TIME
09
11
13
15
17
19
21
23
01
03
05
07
12-14 JULY 1965
16-17 JULY 1965
AVERAGE CONCENTRATION OF FLOATABLES
INFLUENT 5.2 mg/l
EFFLUENT 2.9 mg/l
INFLUENT 4.9 mg/l
EFFLUENT 2.2 mg/l

Reasonably good material balances were obtained for the hexane extractable material during the two periods, whereas the floatable balances were less heartening. During both periods more floatables were actually removed in skimmings than could be expected by the difference between the amount of floatable material in the influent and in the effluent.

Although no definite answer can be given, there appear to be only two possible explanations for this result. The first would be that the preaeration caused material to float to the surface by means of air bubbles attaching to suspended solids despite the results obtained in the laboratory as described in Chapter V. If this is the case, the floated matter would be expected to contain a significant amount of non-grease material carried to the surface by the attached bubbles. Analysis of the floated material revealed (Table D-III) that it consisted almost entirely in grease, 98 percent and 93 percent, respectively, for the two surveys compared to only about 50 percent grease in the dry skimmings collected by vacuum flotation technique.

TABLE VI-II

FLOATABLE MATERIAL BALANCE

Richmond Water Pollution Control Plant Survey

Time	Type of Flow	Total Amount of Floatable Material During Period Dried at 40° C		Difference
13 July 0900	Influent	270 lbs *)	270 lbs	
	Effluent	135 lbs		890 lbs
14 July	Skimmings	1,010 lbs	1,160 lbs	
0700	Sludge	15 lbs		
16 July 0900	Influent	295 lbs	295 lbs	
	Effluent	132 lbs		230 lbs
17 July	Skimmings	372 lbs	525 lbs	
0700	Sludge	21 lbs		

*) For details see Appendix D.

The second explanation is than the additional floated material was caused by the mechanical effect of shear in the comminution and by the mechanical effect of the mixing in the preaeration basin. This explanation is also supported by the observation made during the development of the floatables test where it was found that rapid mixing (100 RPM in the funnel) resulted in liberation of additional grease particles from the suspended solids.

TABLE VI-III

HEXANE EXTRACTABLE MATERIAL - MASS BALANCE

Richmond Water Pollution Control Plant Survey

Time	Type of Flow	Concentration of HEM*) in Composites	Total Flow or Amount During Period	Weight of HEM*) lbs.	
13 July 0900	Influent	81.4 mg/l	6.81 MGD	4,623**)	4,623 lbs.
	Effluent	47.9 mg/l	6.85 MGD	2,736	4,396 lbs.
14 July	Sludge	6,036 mg/l	0.0133 MGD	670	
0700	Skimmings	50%	1980 lbs	990	
16 July 0900	Influent	92.9 mg/l	6.82 MGD	5,284	5,284 lbs.
	Effluent	35.3 mg/l	6.82 MGD	2,007	3,417 lbs.
17 July	Sludge	6,051 mg/l	0.0211 MGD	1,065	
0700	Skimmings	43.7%	790 lbs	345	

*)Hexane Extractable Material.

**)mg/l x MGD x 8.34 = Total weight in pounds.

TABLE VI-IV

VARIATION IN HEXANE EXTRACTABLE MATERIAL CONCENTRATION WITH TIME

Date & Time of Collection July 1965		HEM+) Conc. mg/l	Flow Rate MGD	Weight of HEM lbs.
13	0900	74.0	9.5	488.6*)
13	1100	81.8	9.4	535.1
13	1300	93.1	9.2	595.3
13	1500	91.4	7.0	444.7
13	1700	155.5	6.1	659.2
13	1900	82.5	6.5	372.7
13	2100	85.6	8.1	481.9
13	2300	39.0	7.0	189.7
14	0100	59.5	5.0	206.8
14	0300	53.5	4.5	167.3
14	0500	44.4	3.8	117.3
14	0700	20.3	5.5	77.6
Total Amount of HEM				4,336.2 lbs

Total Amount of HEM determined from Composite Sample was 4,622.8 lbs for the 24-hour period. (See Table VI-III).

+)HEM = Hexane Extractable Material.

*)Pounds of HEM in 2-hour period = mg/l x MGD x $\frac{2}{24}$ x 834

= mg/l x MGD x 0.695

VACUUM FLOTATION

Plant Description

The South San Francisco-San Bruno (SSF-SB) Water Pollution Control Plant is an intermediate treatment plant with an average design flow of 12.9 MGD. The flow diagram for the vacuum flotation system is shown in Figure VI-4 and the design characteristics pertinent to the vacuum flotation system are presented in Table VI-V.

The material floating in the vacuators is transferred to a sump, and from there it is pumped to the digesting system. The vacuators are also provided with a system for removal of settleable solids which are also pumped to the digestion system.

TABLE VI-V

PERTINENT DESIGN CHARACTERISTICS FOR
SOUTH SAN FRANCISCO-SAN BRUNO WATER POLLUTION CONTROL PLANT

Flow:		
San Bruno	Domestic & Commercial	3.69 MGD
South S.F.	Domestic & Commercial	4.24 MGD
South S.F.	Industrial	5.01 MGD
Total average dry weather flow		12.94 MGD
Vacuators, Dorr-Oliver		
Diameter		50 feet
Water depth		10.25 feet
Overflow at design flow		3000 G/D/SF
Detention Time		34 minutes

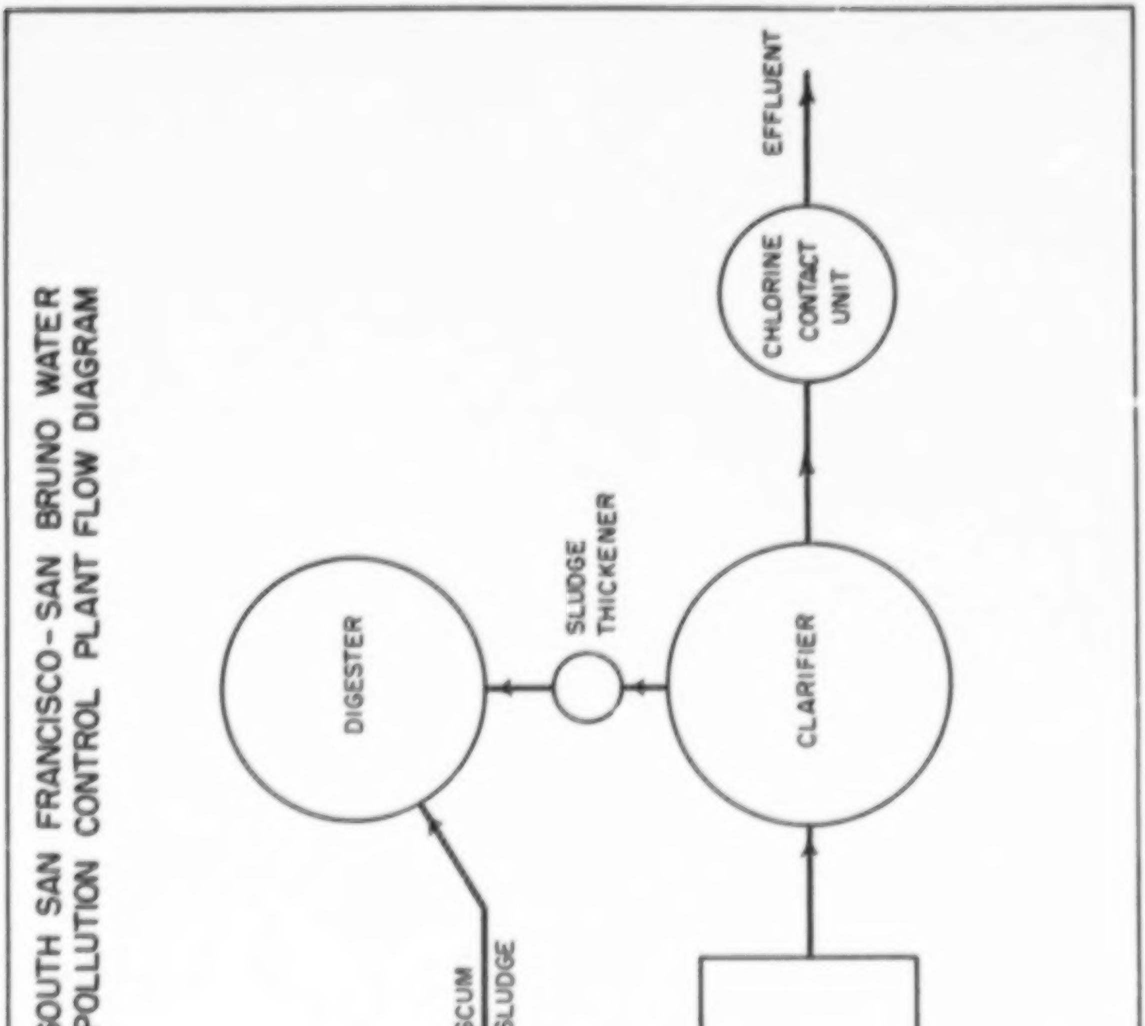
Sampling and Analytical Program

A sampling program similar to that described in the previous section was used with the following two changes: The amount of floated material was estimated from the rate of pumping of the two plunger type skimming pumps which had a combined pumping rate of 105 gallons per minute. These pumps operated an estimated 8 minutes per hour according to the plant operators, corresponding to 20,000 gpd or 0.02 MGD. (The pumps were adjusted to pump 17 minutes per hour during the survey but drained the sump in approximately 8 minutes), and secondly: Since only three samples could be analyzed for floatable material during each two-hour period, samples of the skimmings and samples of the sludge were analyzed alternating every two hours.

Sampling Results

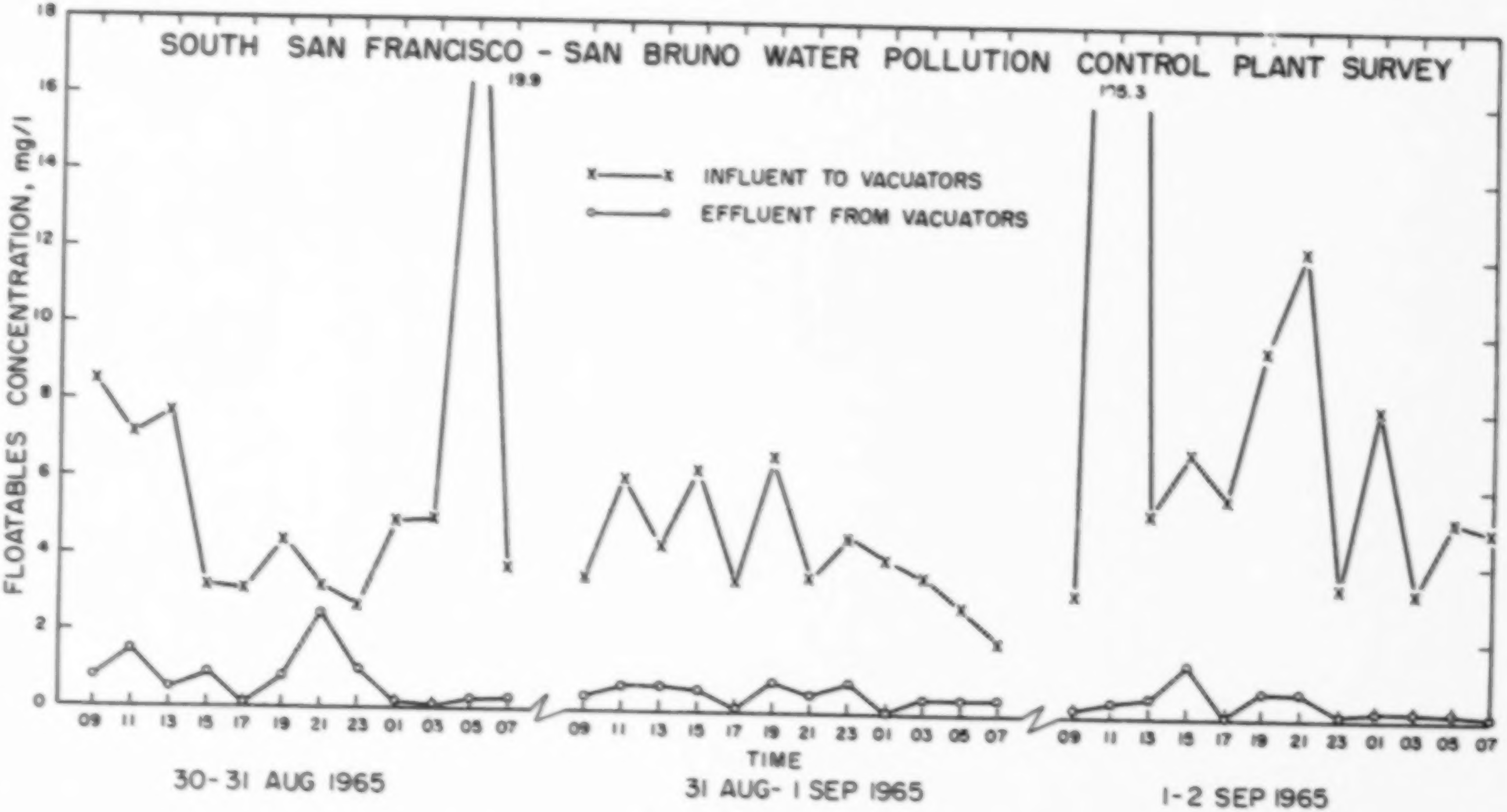
The overall results of the sampling are shown in Figure VI-5 and in Tables VI-VI and VI-VII; the detailed results are presented in Appendix E. The average concentrations of floatable material in the influent were 6.1 mg/l, 4.2 mg/l and 7.8 mg/l during the three survey periods, and the corresponding average concentrations in the effluent were 0.75 mg/l, 0.48 mg/l and 0.39 mg/l.

FIGURE VI-4



SOUTH SAN FRANCISCO-SAN BRUNO WATER POLLUTION CONTROL PLANT FLOW DIAGRAM

RAW SEWAGE INFLOW
GRIT CHAMBER
A
VACUATOR
B
D
SCUM
SLUDGE
C
DIGESTER
SLUDGE THICKENER
AERATION TANK
CLARIFIER
CHLORINE CONTACT UNIT
EFFLUENT
⊗ SAMPLING POINT



AVERAGE CONCENTRATION OF DRY FLOATABLES AT 40 °C

INFLUENT	6.1 mg/l	INFLUENT	4.2 mg/l	INFLUENT	7.8 mg/l
EFFLUENT	0.75 mg/l	EFFLUENT	0.48 mg/l	EFFLUENT	0.39 mg/l
SKIMMINGS	2800 mg/l	SKIMMINGS	2500 mg/l	SKIMMINGS	5760 mg/l

FIGURE VI-5

TABLE VI-VI

FLOATABLE MATERIAL-MASS BALANCE

South San Francisco-San Bruno Water Pollution
Control Plant Survey

Time	Type of Flow	Total Amount of Floatable Material	
30 Aug 0900	Influent	392 lbs.	392 lbs.
31 Aug 0700	Effluent Skimmings	59 lbs. 467 lbs	526 lbs.
31 Aug 0900	Influent	287 lbs.	287 lbs.
01 Sep 0700	Effluent Skimmings	33 lbs. 415 lbs.	448 lbs.
02 Sep 0900	Influent	576 lbs.	576 lbs.
03 Sep 0700	Effluent Skimmings	29 lbs. 961 lbs.	990 lbs.

TABLE VI-VII

HEXANE EXTRACT MATERIAL - MASS BALANCE

South San Francisco-San Bruno Water Pollution Control Plant Survey

Time	Type of Flow	Concentration of HEM+) in Composites	Total Flow or Amount During Period	Weight of HEM+)	
30 Aug 0900	Influent	193.8 mg/l	8.1 MG	13,092 lbs.	13,092 lbs.
31 Aug 0700	Effluent	114.0 mg/l	8.1 MG	7,701 lbs.	12,521 lbs.
	Skimmings	28,900 mg/l	0.02 MG	4,820 lbs.	
31 Aug 0900	Influent	138.1 mg/l	7.5 MG	8,632 lbs.	8,632 lbs.
1 Sep 0700	Effluent	81.8 mg/l	7.5 MG	5,117 lbs.	11,055 lbs.
	Skimmings	35,575 mg/l	0.02 MG	5,938 lbs.	
2 Sep 0900	Influent	221.8 mg/l	7.6 MG	14,071 lbs.	14,071 lbs.
3 Sep 0700	Effluent	109.7 mg/l	7.6 MG	6,972 lbs.	12,638 lbs.
	Skimmings	33,875 mg/l	0.02 MG	5,655 lbs.	

+) Hexane extractable material = mg/l x MG x 8.34

The three composite samples of skimmings were analyzed for percent moisture and percent hexane extractable material. The results are reported in Table VI-VIII and show that the skimmings contain between 8.8 percent and 9.4 percent dry material. The hexane extractable material varied between 33 percent and 41 percent of the total dry skimmings as compared to 93 and 98 percent at the Richmond Plant. Furthermore, the amount of true floatable material in the skimming was determined every four hours by resuspending 50 ml of the skimmings in 3 liters of tap water and determining the floatable material. The average amount of floatable material in the skimmings was 2.80 g/l, 2.49 g/l and 5.76 g/l (Table E-I) during the three survey periods or less than five percent of the dry material in total skimmings.

As for the Richmond Survey, a material balance was attempted for the floatable material and for the hexane extractable material over the vacuators. The results are presented in Tables VI-VI and VI-VII and show a reasonable balance for the hexane extractable material whereas the balance for the floatable material is less good. The major reason for the lack of balance is believed to be the inaccuracy in the determination of the total amount of skimmings based upon the estimated pump rate.

ADDITIONAL PLANT SURVEY

In addition to the two surveys described above, a program of sampling was conducted by the Los Angeles County Sanitation Districts at the Joint Water Pollution Control Plant. The program consisted in collecting daily samples from the effluent of their two sets of primary flotation systems and determine the concentration of floatable material. The objective of this sampling program was to gain additional information about the floatable determination method and about the concentration of floatable material in primary effluents from another area. The results are reported in Table VI-IX and show the average concentration of floatable material to be 1.3 mg/l and 1.7 mg/l respectively for the two primary systems.

Comparison Among The Three Treatment Plants

A summary of the concentration of floatable material in the effluent from the three treatment plants investigated is presented in Table VI-X. The average concentration from the two primary plants employing gravity flotation was 2.2 mg/l and 1.5 mg/l respectively compared to a concentration of 0.6 mg/l in the effluent from the vacuum flotation system. Statistical analysis of the results showed that there is no significant difference between the individual series of measurements for each of the three plants, whereas the difference between each of the plants is significant at the 95 percent confidence level.

DISCUSSION

The most striking result obtained from these treatment plant surveys is the relatively small difference found between the amount of floatable material in raw sewage and in primary effluents and between effluents from different types of primary plants.

The limited amount of data collected here indicates that primary treatment removes only between 50 and 80 percent of the total amount of floatable material (as compared to about 100% removal of settleable solids);

TABLE VI-VIII

MOISTURE CONTENT OF FLOATED MATERIAL COLLECTED DURING SSF-SB WATER POLLUTION CONTROL PLANT SURVEY

August-September 1965

Period of Compositing	Initial Net Weight of Sample (grams)	Dry Weight at 40° C (grams)	Dry Material at 40° C (percent)	Dry Weight at 105° C (grams)	Percent HEM*) of Wet Material	Percent HEM*) of Dry (40°C) Material
30 Aug 0900 | 31 Aug 0700	402.0	35.0	8.7%	33.0	2.89%	33%
31 Aug 0900 | 1 Sep 0700	29.82	2.62	8.8%	2.62	3.56%	41%
2 Sep 0900 | 3 Sep 0700	30.70	2.90	9.4%	2.82	3.39%	36%

*)Hexane extractable material determined according to Reference 1 by extracting mixture of scum and $MgSO_4$, H_2O.

TABLE VI-IX

JOINT WATER POLLUTION CONTROL PLANT
LOS ANGELES COUNTY

	Floatable Material in Primary Effluent	
Date of Sampling	Effluent Channel #2	Effluent Channel #3
June 15, 1965	9.8 mg/l	2.3 mg/l
June 16, 1965	33.2 "	1.5 "
June 17, 1965	9.3 "	3.3 "
June 18, 1965	6.4 "	2.0 "
June 21, 1965	23.0 "	3.0 "
June 22, 1965	6.2 "	1.0 "
June 23, 1965	10.8 "	2.2 "
June 24, 1965	3.9 "	1.5 "
July 6, 1965	* 1.3 "	1.3 "
July 7, 1965	1.1 "	1.0 "
July 9, 1965	1.7 "	3.2 "
July 12, 1965	1.2 "	1.1 "
July 14, 1965	1.8 "	0.9 "
July 16, 1965	0.9 "	1.5 "
August 5, 1965	1.1 "	0.8 "
August 6, 1965	1.4 "	0.7 "
August 9, 1965	1.3 "	0.9 "
August 10, 1965	1.0 "	1.5 "
Average	1.3 mg/l	Average 1.7 mg/l

*) Sampling location for Effluent Channel #2 changed, early data not included in average.

TABLE VI-X

COMPARISON AMONG CONCENTRATIONS OF FLOATABLE MATERIAL IN PRIMARY EFFLUENT FROM THREE PRIMARY TREATMENT PLANTS

	RICHMOND WPCP		SOUTH SAN FRANCISCO-SAN BRUNO WPCP			LOS ANGELES COUNTY JWPCP	
	Gravity Flotation		Vacuum Flotation			Gravity Flotation	
	First 24 hr period	Second 24 hr period	First 24 hr period	Second 24 hr period	Third 24 hr period	Effluent Channel No. 2	Effluent Channel No. 3
Average Floatables	2.2 mg/l	2.2 mg/l	0.8 mg/l	0.5 mg/l	0.4 mg/l	1.3 mg/l	1.7 mg/l
Number of Measurements	12	12	12	12	12	10	18
Standard Deviation	1.4 mg/l	1.4 mg/l	0.7 mg/l	0.2 mg/l	0.4 mg/l	0.3 mg/l	0.9 mg/l
Average Floatables	2.2 mg/l		0.6 mg/l			1.5 mg/l	
Number of Measurements	24		36			28	
Standard Deviation	1.4 mg/l		0.5 mg/l			0.7 mg/l	

nevertheless, primary treatment has been generally effective in preventing the build-up of objectionable scum deposits in rivers and along beaches.

An explanation may be that primary treatment preferentially removes the larger particles of floatable material which are easily recognized as of waste origin, and the smaller particles are biologically decomposed at a fairly high rate, thus preventing an aesthetic nuisance. Another possible explanation is that discharge of raw sewage may result in the formation of sludge banks from which additional floatable material may later be liberated by mechanical or bacteriological action.

The results of this study indicate a relatively small numerical difference between the amount of floatable material in effluents from gravity and vacuum flotation systems (between one and two mg/l). At the present time it is not known whether this difference is large enough to warrant the use of vacuum flotation rather than well designed and operated gravity flotation for treatment before disposal into the marine environment. Nevertheless it should be noted that the floatable material is concentrated on the receiving water surface and floatable material from a large volume of discharge may be concentrated by wind and currents on a limited surface beach area. If this is the case, a reduction in floatables from 2.2 mg/l to 0.6 mg/l will correspond to a 75 percent reduction of sewage floatables on the beach.

Investigations planned for the forthcoming year will be directed to the evaluation and improvement of conventional gravity flotation systems as well as to the evaluation and development of vacuum flotation systems for the removal of floatable material.

SUMMARY AND CONCLUSIONS

1. The concentration of floatable material in primary effluent was determined under plant conditions for plants using ordinary gravity flotation and a Dorr vacuum floatator.

2. The reductions in floatables at the plant employing gravity flotation for two field test periods were from 5.2 to 2.9 mg/l and from 4.9 to 2.2 mg/l. The reductions with the Dorr vacuum process for three test periods were from 6.1 to 0.75 mg/l, from 4.2 to 0.48 mg/l, and from 3.8 to 0.39 mg/l.

3. Although materials balances were not made, the data indicate that the skimmings from gravity flotation contain a higher percentage of hexane extractable material than skimmings from Dorr vacuum flotation, presumably because the flotation process brings some settleable solids to the surface.

4. One of the significant findings of this study is that the magnitude of floatables in raw sewage at the plants studied (ranging from an average of 5.2 to 4.9 mg/l) is apparently not vastly different from the magnitude in primary effluents (ranging from an average of 2.9 to 2.2 mg/l); nevertheless this level of reduction apparently is of great significance with respect to effects on the receiving environment, presumably because of the effects at the surface when the floatables dispersed in the sewage volume become concentrated at the surface. These relationships merit detailed investigation.

VII. DETERMINATION OF FLOATABLE MATERIAL IN THE OCEAN

INTRODUCTION

In the discharge of sewage into the ocean, coliform and floatables are the two most important indices of beach pollution. The collection of representative samples of floatable material, therefore, is an essential step in the monitoring or evaluation of submarine disposal of sewage.

Despite many attempts in recent years, no simple and dependable method of sampling has been devised. In a study by Merz for the California State Water Pollution Control Board (Reference 24), several types of stationary floats were constructed and tested. The floats varied in form and methods of anchoring. They were filled with absorption pads through which known amounts of water were pumped. Oil and grease were retained by the pad and subsequently extracted with a suitable solvent. Although this type of sampler worked mechanically, several drawbacks have prevented its widespread use. Some of the drawbacks are:

1. Cost of construction, operation and maintenance is high.
2. The sampler must be located near power supply but inaccessible to the public.
3. The sampler pumps water not only from the surface but also from underneath the surface, and it is not possible to quantitate the grease and oil extracted in terms of weight per unit surface area.
4. The method will not measure floatables of non-grease origin.

One other method commonly used in the field is the collection of floatables from the ocean surface by dipping a bucket and analyzing the sample for grease and oil. This method is inadequate because the areal concentration of floatables is generally very low, and the result cannot be expressed on the basis of surface area. Also, the disturbance of the water surface at the time of dipping may lead to erroneous results.

An ideal sampler for the collection of floatables should satisfy the following requirements:

1. It should collect the sample from the surface only.
2. The area of sampling should be known.
3. It should be simple and dependable.

Floatable Sampling Apparatus

In the first annual report, two stationary samples were described. The first one was a triangular float and the other was an anchored "V" shaped float. Neither of these two samples were very successful.

The third sampler designed during this study is a surface skimmer provided with a large net. Instead of relying on a pump or the ocean current for the throughput of sea water, it is to be trawled on the water surface by a boat. In order to have the sampler encounter undisturbed water surface, it is pulled away from the wake of the trawling boat by means of a paravane (or "otter board").

The sketch of the sampler and otter board are shown in Figures VII-1 and VII-2. The prototype sampler consists in a wood frame, floats, and a net. The floats are attached in such a way that the sampler can maintain horizontal position while skimming a surface layer down to about 6 inches depth.

To impart a horizontal force in a right angle to the direction of the boat's movement, a 3 ft x 4 ft otter board was tested, initially. With proper floats and weights the planar otter board functioned well. However, when the waves were higher than two-three feet, the otter board tended to lose its stability. To resolve this problem, a trawling cone which was devised and patented by Fynbo (Reference 25) was adapted for use instead, with excellent results.

Sampling Net

Several different types of surplus nets were tested during this investigation. The first was a Dacron net with 0.3 mm openings. During the first field testing in the vicinity of the Watsonville, California submarine outfall the net clogged and caused excessive drag. This made it difficult to keep the samples away from the wake of the boat and also causes deflection of surface water around the sampler.

Three methods were considered which might minimize this problem:

1. Selecting a net with a larger area.

2. Frequent change of net.

3. Using a net with a large mesh size.

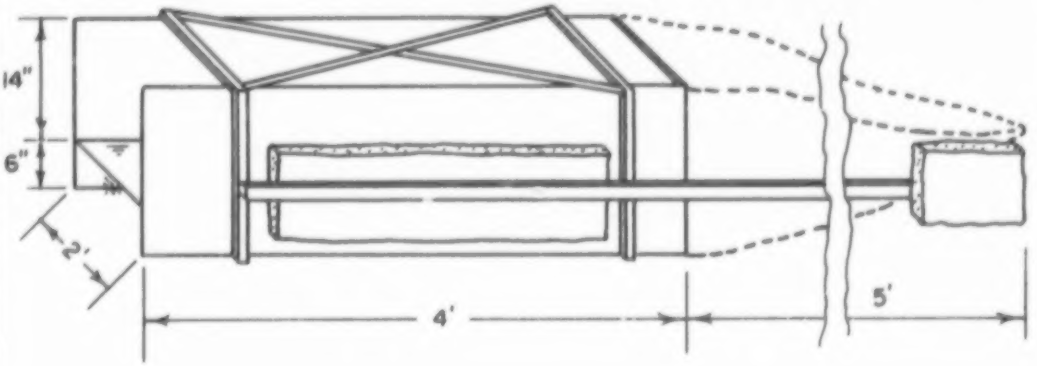
A combination of 2. and 3. was selected for use in subsequent samplings. Several nets were made of cotton with 0.5 mm openings. The nets were 6 ft long, conically shaped and had a 1.5 feet x 2 feet opening. During a four mile test in skimming a place outside Los Angeles Harbor, no significant clogging was observed and the sampler could be kept outside the wake of the boat without any observable deflection of the flow lines around the sampler.

The Efficiency of Collection by the Sampler

In order to estimate the fraction of floatables retained by the net with the 0.5 mm opening compared to the total floatable material, a test was conducted with effluent sewage from a primary settling basin. The result showed that 68% of the total weight of floatables was retained by the net while 32% of the total passed through the filter net. The floatables were determined as described in Appendix B.

FIGURE VII-1

SKETCH OF FLOATABLES SAMPLER



SKETCH OF CONE SHAPED "OTTER BOARD"

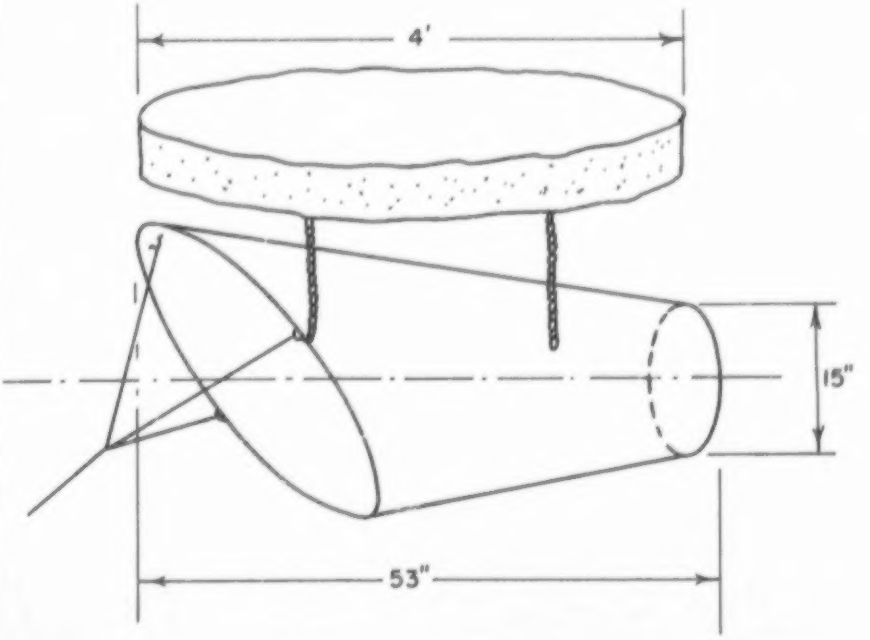
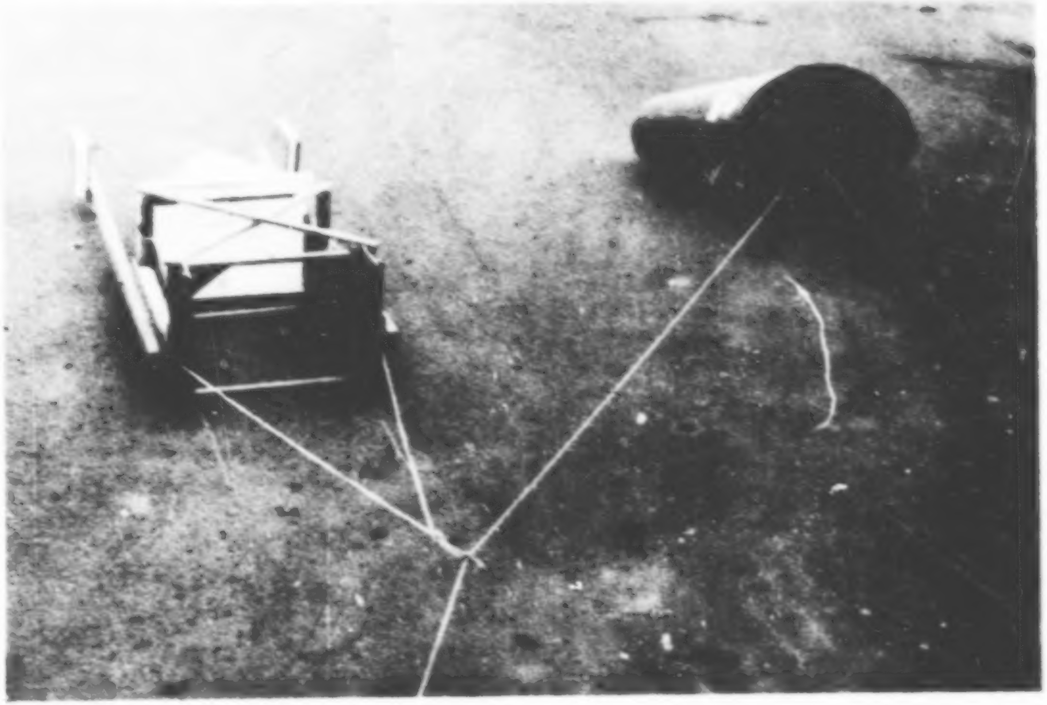


FIGURE VII-2

FLOATABLES SAMPLER

(A float attached to the "cone shaped otter board" not shown)



Also, a test was made to find out whether the floatables in the path of the sampler will all pass through the openings of the sampler or be deflected. A special attachment was made to the sampler so that a uniform spread of small plastic pellets (3.0 mm in diameter) could be made during the time of skimming. A known number of polyethylene pellets were spread uniformly in six feet width, at four feet in front of the sampler (the sampler is two feet wide). By comparing the number of plastic pellets collected with the initial spread of the plastic pellets, the efficiency of collection was found to be 105%. The reason for collection of more than 100% of plastic pellets in the path of the sampler may be due to the water which hit the float at the side of the sampler and splashed over the side wall (6" high) into the sampler. This phenomenon could be observed during the test. To eliminate this possible cause, the height of the side wall was increased an additional eight inches. However, no further test was made to check the result of the correction.

Operation of the Sampler

In order to determine the concentration of floatables per unit area, the surface area covered by the sampling device must be measured.

The distance of surface travel by the sampler in relation to a hypothetical quiescent body of water (this means that the velocity of current has to be accounted for) may be found out by one of the two methods described below:

1. Sampling a known distance twice, in opposite directions or along a closed curve around an outfall will allow determination of the distance of skimming regardless of the current velocity provided that the current velocity does not change greatly during the period of sampling.
2. The use of a cumulative current meter (or odometer) attached to the side of the sampler will record the distance (relative to the quiescent body of water) of skimming covered by the sampler. This method does not necessitate the assumption of constant current velocity as above.

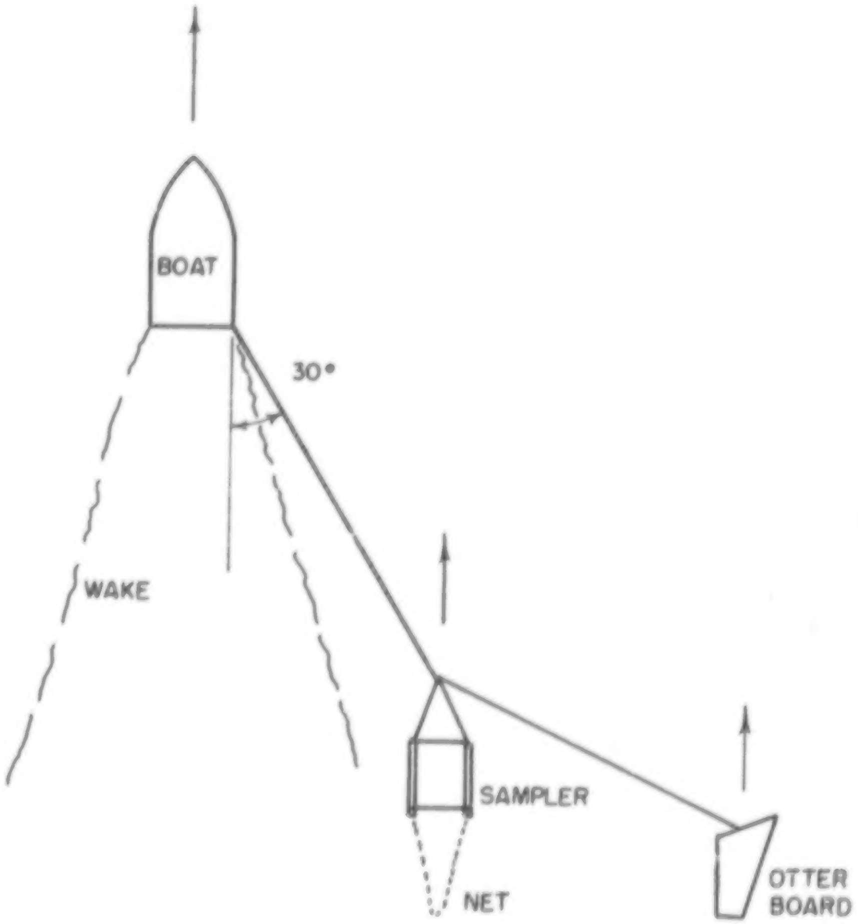
Considering the time and expenses which would be involved in the adaption of a cumulative current meter it was decided to use the method of sampling along a closed curve around the outfall during this preliminary evaluation of the sampler.

The connection of the sampler and otter board to the boat was made as shown in Figs. VII-3 and VII-4. The sampler is best operated at a speed between two and three knots. The wake of the boat used in this study spread out in a 15° angle, while the angle between the boat direction and the pulling line was about 30°.

Results of Floatables Sampling

Upon completion of the preliminary tests in the San Francisco Bay, a program of sampling was carried out in cooperation with the Los Angeles County Sanitation Districts, sampling in the vicinity of the submarine outfalls from the Districts' Joint Water Pollution Control Plant.

SKETCH OF THE SAMPLER TRAWLING METHOD


BOAT
30°
WAKE
SAMPLER
NET
OTTER BOARD

SURFACE TRAWL WITH SAMPLER

The sampler was dragged along the four sides of a square surrounding the outfalls. Each of the four sides was approximately one mile long. After one complete round along the four sides of the square, the collected floatables were brought back to the laboratory in the net and transferred quantitatively to a Flotation Funnel. The collected sample in the net consisted of fish eggs and slimes as well as grease balls. Therefore, the sample was suspended into three liters of water and mixed and settled as described in Steps 1 through 4 in Appendix B. This was done with the purpose of separating fish eggs, plankton, and other non-floatable material, which was incidental collection, from true floatable material. The floatable material was then dried at 40° C until constant weight was obtained, and its content was determined.

In the procedure of suspending the collected sample in the Flotation Funnel, tap water was used first. A significant portion of the sample was observed settling at the bottom of the funnel. Suspecting that this may have been caused by the difference in density between the sea water and tap water, the settled portion was resuspended into NaCl solution simulating sea water. Most of the previously settled portion floated in the NaCl solution, again.

The results of laboratory analysis is shown in Table VII-I.

Analysis of the Results

Comparing the total floatables concentration during the two days of sampling, 7th and 8th September, the variation is very significant. This result is in good agreement with the visual observation of the ocean surface during the two days of sampling. It will be interesting to compare the fluctuation of floatables with other pertaining factors such as wind direction and velocity, current velocity, water temperature, etc.

The increase in the amount of floating material with the increasing density of the media is shown in Table VII-I. The 56 percent increase in floatables of the second day's sample should be taken with reservation because the volume of floatables was very large (about 500 ml) and may have prevented complete separation of floatables from lime in the initial flotation procedure. The content of salt, however, is shown to affect floatability and the relative increase is of the same order of magnitude as found in Chapter IV. In the future work of this type, the sea water collected in the sampling area will therefore be used in floatables test.

In the first day's sampling, two nets were used. The first net covered about 1/4 of the total area along the nearshore side of the square, and the second net covered the rest. The result showed that the surface concentration at the nearshore side was only 1.1 mg/100 m^2 while the remaining three sides of the square had a floatables concentration of 17.3 mg/100 m^2. The areal distribution of floatables can therefore be determined by sampling several small sections, and this kind of data may prove significant when correlated with other oceanographic data.

TABLE VII-I

AREAL CONCENTRATION OF FLOATABLES AND ITS GREASE CONTENT NEAR LOS ANGELES COUNTY SANITATION DISTRICTS OUTFALLS

Distance Trawled = 25,650 ft.

Area covered by the sampler = 51,300 sf = 4,770 square meters

Date	7 Sept 1965	8 Sept 1965
Floatables		
Floated by Fresh Water (mg/100 m^2)*	10.0	1,072.
Floated by Salt Water (mg/100 m^2)	13.2	1,675
Percent Increase of Floatables by Salt Water	32%	56%
Percent of Total	24%	36%
Grease Content**		
Floated by Fresh Water	61.5%	79.6%
Floated by Salt Water	61.2%	76.1%

*1.0 mg/100 m^2 = 0.93 mg/1000 sf.

**This represents only the free fatty acid portion. Soaps were not included in the grease content, but may account for another 10 percent of the total floatable material, based upon analysis of a part of the sample from 8 September.

SUMMARY

1. A sampler has been developed for quantitative collecting of floatable material on the ocean surface.
2. The sampler permits quantification of floatable material in mass per unit area of surface.
3. Sampling in the vicinity of existing submarine ocean outfalls have showed good correlation between visual observation of floatable material and the amount of material collected with the floatables sampler.

VIII. SUMMARY AND CONCLUSIONS

HEXANE EXTRACTION METHOD

1. A liquid-liquid extraction method for the determination of Hexane Extractable Material in sewage has been developed using the direct extraction of a 1-liter sample.

2. Extraction of fat and fatty acid standards showed an average recovery of 98.5 percent for samples containing from 6 mg to 100 mg fat per liter. The standard deviation of the method, as determined on homogenized raw sewage, was about 1.0 mg/l for samples containing from 30 mg/l to 100 mg/l of Hexane Extractable Material, corresponding to a coefficient of variation between 3 percent and 1 percent in this range.

3. Factors affecting the extraction efficiency, acidification, preheating, duration of extraction, and evaporation of solvent were evaluated, and it is concluded that the most reliable extraction results are obtained when samples are acidified and preheated at 72-75°C for one hour followed by three hours of extraction. The solvent evaporation procedure was found to consist in evaporation to dryness at room temperature, followed by drying for five minutes at 85°C. The final method is described in detail in Appendix A.

DETERMINATION OF FLOATABLE MATERIAL IN DOMESTIC SEWAGE

1. A method for determination of floatable material in domestic sewage has been developed using a special Teflon coated Flotation Funnel. The method includes material which floats only after contact with sea water.

2. The effects of mixing time, flotation time, and sea water addition were investigated in the following levels:

   Mixing: 15, 30, and 60 minutes.

   Flotation: 15, 30, and 60 minutes.

   Sea Water Dilution: No dilution and "infinite" dilution.

   Only sea water dilution was found to be significant, and this only for wastes containing low concentrations of floatable material.

3. The composition of the potential floatable material has not been determined; this is being considered for the third project year.

4. The coefficient of variation and accuracy of the test developed varied from 5.7 percent and 96 percent recovery of floatable material for a sewage with 49 mg floatable material per liter to 20 percent and 92 percent recovery for sewage with 1.0 mg floatable material per liter.

LABORATORY EVALUATION OF TREATMENT METHODS

1. Prolonged flotation times will enhance the removal of floatable material, whereas aeration and the addition of up to 30 percent sea water appear to be without effect.

2. Based on laboratory tests, pressure aeration appears to increase the amount of floated material by as much as ten times for raw sewage and about three times for settled sewage compared to gravity flotation based upon laboratory results.

3. The laboratory data indicate that promising treatment methods for marine disposal are pressure flotation of raw sewage, improved gravity retention focused on improving floatable solids removal, and gravity retention followed by pressure flotation of the effluent.

CONCENTRATION OF FLOATABLE MATERIAL IN DOMESTIC SEWAGE

1. The concentration of floatable material in primary effluent was determined under plant conditions for plants using ordinary gravity flotation and a Dorr vacuum floatator.

2. The reductions in floatables at the plant employing gravity flotation for two field test periods were from 5.2 to 2.9 mg/l and from 4.9 to 2.2 mg/l. The reductions with the Dorr vacuum process for three test periods were from 6.1 to 0.75 mg/l, from 4.2 to 0.48 mg/l, and from 3.8 to 0.39 mg/l.

3. Although materials balances were not made, the data indicate that the skimmings from gravity flotation contain a higher percentage of hexane extractable material than skimmings from Dorr vacuum flotation, presumably because the flotation process brings some settleable solids to the surface.

4. One of the significant findings of this study is that the magnitude of floatables in raw sewage at the plants studies (ranging from an average of 5.2 to 4.9 mg/l) is apparently not vastly different from the magnitude in primary effluents (ranging from an average of 2.9 to 2.2 mg/l); nevertheless this level of reduction apparently is of great significance with respect to effects on the receiving environment, presumably because of the effects at the surface when the floatables dispersed in the sewage volume become concentrated at the surface. These relationships merit detailed investigation.

DETERMINATION OF FLOATABLE MATERIAL IN THE OCEAN

1. A sampler has been developed for quantitative collecting of floatable material on the ocean surface.

2. The sampler permits quantification of floatable material in mass per unit area of surface.

3. Sampling in the vicinity of existing submarine ocean outfalls have showed good correlation between visual observation of floatable material and the amount of material collected with the floatables sampler.

IX. RECOMMENDED INVESTIGATION FOR THE THIRD PROJECT YEAR

INTRODUCTION

During the first two years of work on this project, a reliable method for the measurement of floatable material has been developed, and preliminary testing of possible treatment methods were initiated. Laboratory results on floatable matter removal were obtained using biological treatment methods, gravity flotation and vacuum flotation.

Based upon these results, and upon field evaluation of existing treatment units, it has been concluded that the most promising treatment methods for the removal of floatable material from a waste before discharge into the marine environment are either to use improved gravity flotation systems or vacuum flotation systems.

In addition, a sampler has been developed for collecting floatable material in the ocean, but sufficient data have not yet been collected to permit an evaluation of the accuracy or precision of the sampling device or of the allowable concentrations of floatable material on the ocean surface.

STUDY PLAN

The proposed study plan for the third year will consider the following three areas of investigation.

A. A pilot plant investigation, using pilot or field-scale flotation units of the pressure and/or vacuum flotation type for removal of floatable material from both raw and settled sewage. Attention will be given to the question of removal of settleable solids during the flotation process, since settleable solids may float under these conditions and thus increase the amount of solids to be disposed of on land.

B. A study of the efficiency of removal of floatable material from existing primary sedimentation basins. Special emphasis will be placed upon the efficiency of existing skimming methods since numerous field inspections have indicated that particles may well float in a basin but are not collected by existing skimming devices.

C. Continue the investigation of floatable material in the ocean in the vicinity of existing ocean outfalls to evaluate the accuracy and efficiency of the sampling device and to obtain reliable information (on a basis of mass per surface area) about the allowable concentrations of floatables and eventually a correlation between floatables found in the ocean and effluent characteristics.

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APPENDICES

A. Liquid-Liquid Extraction of Oil and Grease from Sewage

B. Laboratory Determination of Floatable Material in Sewage

C. Analysis of Variance for the Factorial Experiments

D. Floatable Material in Influent and Effluent (Richmond Water Pollution Control Plant)

E. Floatable Material Survey (South San Francisco - San Bruno Water Pollution Control Plant)

APPENDIX A

LIQUID-LIQUID EXTRACTION OF OIL AND GREASE FROM SEWAGE

GENERAL DISCUSSION

Principle: The salts of the fatty acids are first hydrolyzed by acidification and heating for one hour. Oil and grease is then extracted in a continuous extractor, and the residue remaining after evaporation of the solvent is weighed. Compounds volatilized at or below 85° C will be lost.

Interferences: This method is subject to the same interferences as the Soxhlet Extraction Method.

APPARATUS

Sampling Bottle: A 1-liter Pyrex flask provided with a 34/45 standard ground glass joint and a 1000 ml calibration mark.

Water Bath: Water bath which can be adjusted to 72° - 75° C.

Extraction Apparatus: A Pearson extraction apparatus as shown in Figure A-1.

Glass Filter Paper: Whatman GF/c. 4.25 cm.

Magnetic Stirrer: Precision Scientific Catalog No. 65904 with a 1-1/2 inch Teflon coated stirring bar.

Hot Plate

Aluminum Dishes: 5.5 cm, Van Waters & Rogers, Inc. Catalog No. 25433.

REAGENTS

Hydrochloric acid, conc.

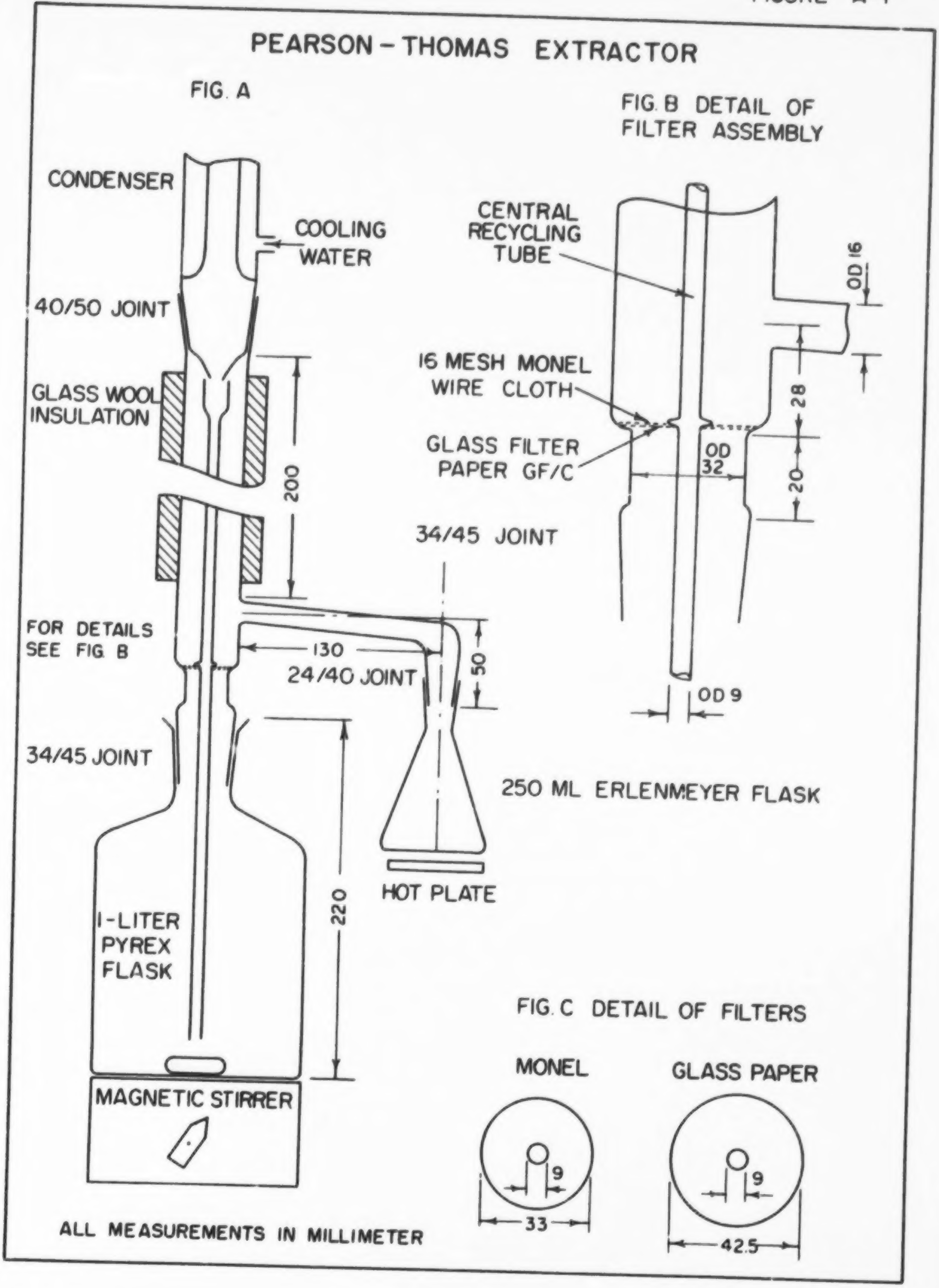
n-Hexane, boiling point 68-69° C, purified grade.

PROCEDURE

Collect a 1-liter volume of sewage in the sampling bottle (which also serves as extraction chamber) and acidify by adding 10 ml conc. HCl. Place the acidified sample in the 72-75° C water bath for one hour. Remove and cool to below 65° C.

Place the Teflon coated magnetic stirring bar in the extraction flask; place the glass filter paper and the monel screen in the cylinder and assemble the extraction apparatus as shown in Figure A-1. Seal the 34/45 joint at the top of the extraction flask with distilled water. Add 150 ml hexane to the Erlenmeyer flask and set the mixer at speed 7 (to obtain a small vortex). Start the extraction and adjust the hot plate so the extraction rate is between 400 ml and 600 ml hexane per hour. (Between 15 minutes and 20 minutes from

FIGURE A-1



PEARSON-THOMAS EXTRACTOR

FIG. A
CONDENSER
COOLING WATER
40/50 JOINT
GLASS WOOL INSULATION
200
FOR DETAILS SEE FIG. B
130
24/40 JOINT
50
34/45 JOINT
34/45 JOINT
250 ML ERLENMEYER FLASK
HOT PLATE
220
1-LITER PYREX FLASK
MAGNETIC STIRRER
FIG. B DETAIL OF FILTER ASSEMBLY
CENTRAL RECYCLING TUBE
OD 16
16 MESH MONEL WIRE CLOTH
28
GLASS FILTER PAPER GF/C
OD 32
20
OD 9
FIG. C DETAIL OF FILTERS
MONEL
GLASS PAPER
9
33
9
42.5
ALL MEASUREMENTS IN MILLIMETER

the time the hexane beings to condense in the top condenser till it starts overflowing back into the Erlenmeyer flask). Extract for three hours, turn off the hot plate, wait five minutes (fire hazard), and remove Erlenmeyer flask.

Evaporate the hexane in the Erlenmeyer flask on a water bath or steam bath till only 10-15 ml are left. Transfer quantitatively to a predired and weighed set of aluminum dishes (rinse three times with hexane from a glass wash bottle). The set of aluminum dishes is made by widening the sides of one dish and placing a similar dish inside the first dish (to prevent losses by creeping). Evaporate the hexane in the dishes to dryness at room temperature. Place dishes in an 85° C oven for 5 minutes, cool in a desiccator for 15 minutes and weigh. Repeat drying procedure until observed weight change is less than 0.5 mg for 5 minutes drying.

CALCULATIONS

mg/l Hexane Extractable Material =

$$\frac{(\text{mg increase in weight of dishes - blank}) \times 1000}{\text{ml sample extracted}}$$

PRECISION AND ACCURACY

An average recovery of 98.5 percent with a standard deviation of 2.3 percent was obtained on Crisco standards. Replicate analyses of three sewage samples containing from 30 mg/l to 100 mg/l Hexane Extractable Material yielded standard deviations between 0.7 mg/l and 1.0 mg/l. The coefficient of variation varies from one to three percent in this range.

SPECIAL PRECAUTIONS

Mixing: The mixing speed is very critical. The speed of the mixer should be such that the refluxing hexane is dispersed in small droplets throughout the sample being extracted and a small vortex created. The speed is best adjusted when extracting a clear water sample.

Blanks: The purified hexane varies from batch to batch. A duplicate blank determination should be made on each batch received, and the average subtracted from the extraction results. Since the blank values are relatively small (0.5 mg/150 ml hexane to 3.0 mg/150 ml hexane) and easily corrected for, it will, in most cases, be unnecessary to use the expensive analytical grade hexane.

Rising Sludge: Sludge may rise in some samples and clog the glass filter paper. This occurs mainly in samples containing a high concentration of suspended solids and is most easily prevented by diluting the samples 1:1 with distilled water before preheating. (Too violent mixing will have the same effect).

APPENDIX B

LABORATORY DETERMINATION OF FLOATABLE MATERIAL IN SEWAGE

GENERAL DISCUSSION

Principle: The floatable material in a sewage sample is concentrated on the surface of the sample in a special Teflon coated Flotation Funnel. The remainder of the sample is drained off and the floatable material collected, washed, and weighed on a glass filter paper. The hexane extractable fraction of the floatable material can be determined subsequently by cutting the filter paper into 1 cm x 1 cm pieces and extracting these,described in Appendix A.

APPARATUS

Flotation Funnel: A Teflon coated 3-liter Flotation Funnel provided with a 7mm bore Teflon stop cock as shown in Figure B-1. The Flotation Funnel should be provided with a 10 ml, 40 ml, 200 ml, and 3000 ml mark.

Mixer: Variable speed paddle mixer adjustable from 40 RPM to 100 RPM.

Paddle: Teflon coated brass paddle 75 mm x 25 mm.

Filter Holder: Teflon coated Millipore filter holders. Catalog No. XX 10 047 00.

Filter Papers: Whatman GF/c. 5.5 cm

Suction Flask

Vacuum Pump

Oven: Adjusted to between 35° C and 40° C.

Cleaning Rod: A 3 mm diameter Teflon coated brass rod 85 cm long.

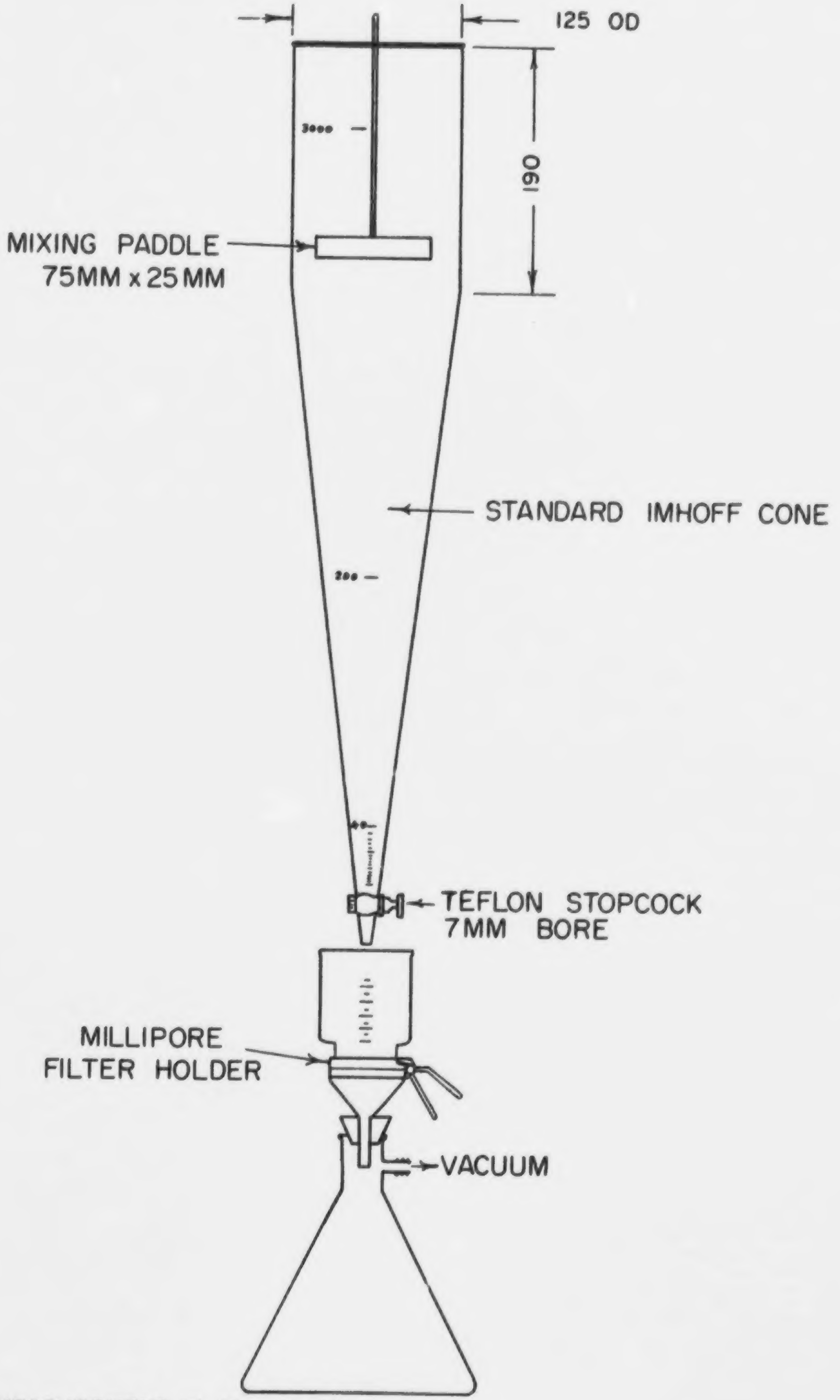
PROCEDURE

Sample Collection and Preparation

Collect an 8-liter sample in a bucket provided with a bottom outlet. Great care must be taken to sample at points where the waste stream is completely mixed. Best results are obtained when the bucket is dipped directly into the waste stream. Transport the 8-liter sample to the laboratory, place a propeller stirrer in the bucket and stir so floatables are thoroughly mixed throughout the whole 8-liter volume. While stirring, transfer three liters through the bottom outlet into the Flotation Funnel. The sample shall be transferred to the Flotation Funnel within two hours after collection to insure that no significant change in the amount of floatable material takes place.

FIGURE B-1

FLOTATION FUNNEL


125 OD
190
MIXING PADDLE 75MM x 25MM
200
STANDARD IMHOFF CONE
TEFLON STOPCOCK 7MM BORE
MILLIPORE FILTER HOLDER
VACUUM

ALL MEASUREMENT IN MILLIMETER

Place the Flotation Funnel in a rack and fasten securely to prevent even slight movements.

Conduct the test at the same temperature as that of the receiving body of water.

Correction for Density and for Concentration Effects

When a waste to be analyzed is being discharged into a receiving water with a density and ion concentration different from that of waste itself, the density and ion concentration of the waste should be adjusted to that of the receiving water. In the frequent case where the receiving water is ocean water, the density adjustment should be done in the following manner: 1.5 l sample is placed in the Flotation Funnel and 1.5 l filtered sea water for the receiving area added together with mixture of 39.8 NaCl, 8.0 g $MgCl_2$-$6H_2O$, 10.1 g $MgSO_4$-$7H_2O$, and 2.3 g $CaCl_2$-$2H_2O$. The final mixture will then contain 1.5 l sample in a medium of the approximately same density and ion concentration as sea water.

Mixing and Flotation

Place the paddle mixer in the Flotation Funnel, mix, settle, and discharge as shown in Table B-I. It is important that the surface of the sample in the Flotation Funnel remains undisturbed during the discharge to prevent loss of floatables. The discharge rate shall be 500 ml per minute except for the last 30 ml which shall be discharged dropwise.

Filtration and Weighing

Place a washed, dried, and weighed glass filter paper in the Teflon coated filter holder and filter the last 10 ml with the floatable material. Wash with distilled water. An additional piece of preweighed filter paper may be used to wipe the bottom of the filter holder if necessary.

Dry the filter (and the additional piece if used) at 35° C or 40° C for 1-1/2 hours, place in a desiccator for 15 minutes and weigh. Redry to constant weight.

CALCULATIONS

$$\text{mg/l Floatable Material} = \frac{\text{mg/increase in weight of filter}}{\text{volume of sample in liters}}$$

PRECISION AND ACCURACY

Replicate analysis of sewage samples as shown in Table B-II, showed the following standard deviations and recoveries:

TABLE B-I

MIXING AND FLOTATION SCHEDULE

1. Mix at 40 RPM for 15 minutes.
2. Let settle 5 minutes.
3. Mix at 100 RPM for 1 minute.
4. Let settle 30 minutes.
5. Discharge 2.8 liters at a rate of 500 ml/min. (Use cleaning rod if settled material clogs the stop cock).
6. Wash mixing paddle and sides of the Flotation Funnel with distilled water from a wash bottle until all particulate matter has moved to the bottom of the funnel.
7. Let settle 15 minutes.
8. Discharge down to the 40 ml mark.
9. Let settle 10 minutes.
10. Discharge dropwise to the 10 ml mark.
11. *Add 500 ml distilled water of same temperature as the sample, and repeat steps seven through ten.

*Step 11 can be omitted for samples containing high concentrations of floatable material and low concentrations of suspended solids.

TABLE B-II

PRECISION AND ACCURACY OF THE FLOATABLES DETERMINATION METHOD

Type of Sewage	Avg. Floatables Concentration mg/l	No of Samples	Coef. of Variation	Percent Recovery
Raw*	49. mg/l	5	5.7%	96%
Raw	1.0 mg/l	5	20%	92%
Primary Effluent	2.7 mg/l	5	15%	91%

*) Additional floatable material added from skimmings from a primary tank.

APPENDIX C

ANALYSIS OF VARIANCE FOR THE FACTORIAL EXPERIMENTS

A. SEWAGE WITH LOW CONCENTRATION OF FLOATABLES

TABLE C-I

EXPERIMENT LAY-OUT AND RESULTS

Replicate I

Block a		Block b		Block c	
Code*	mg/l	Code	mg/l	Code	mg/l
(100)	9.8	(200)	1.4	(000)	5.5
(210)	9.9	(010)	4.3	(110)	4.7
(020)	9.7	(120)	5.0	(220)	6.4
(201)	3.9	(001)	2.1	(101)	4.4
(011)	5.7	(111)	1.7	(211)	3.5
(121)	5.4	(221)	1.9	(021)	5.0

Replicate II

Block a		Block b		Block c	
Code	mg/l	Code	mg/l	Code	mg/l
(200)	21.9	(000)	14.5	(100)	11.9
(110)	23.9	(210)	5.2	(010)	6.9
(020)	13.2	(120)	11.5	(220)	9.9
(101)	6.9	(201)	2.6	(001)	6.0
(011)	14.7	(111)	7.1	(211)	13.3
(221)	11.3	(021)	4.9	(121)	7.7

*First figure in code is level of mixing time, second is level of flotation time, and third refers to sea water-salt dilution. For details, see Chapter IV.

TABLE C-II

COMBINED TREATMENT TOTALS FOR REPLICATE I AND REPLICATE II

	S_0			S_1		
	F_0	F_1	F_2	F_0	F_1	F_2
M_0	5.5 14.5 20.0	4.3 6.9 11.2	9.7 13.2 22.9	2.1 6.0 8.1	5.7 14.7 20.4	5.0 4.9 9.9
M_1	9.8 11.9 21.7	4.7 23.9 28.6	5.0 11.5 16.5	4.4 6.9 11.3	1.7 7.1 8.8	5.4 7.7 13.1
M_2	1.4 21.9 23.3	9.9 5.2 15.1	6.4 9.9 16.3	3.9 2.6 6.5	3.5 13.3 16.8	1.9 11.3 13.2

Explanation: Top figure in each box is result from Replicate I, middle figure in each box is result from Replicate II, and bottom figure is the sum of the results from Replicates I and II. For example, the top figure in the box in the upper right corner is the amount of floatables from treatment code (021) in Replicate I and middle figure is result from code (021) in Replicate II.

TABLE C-III

TWO-WAY TABLE

	$S_0 + S_1$				$S_0 - S_1$			
	F_0	F_1	F_2	Total	F_0	F_1	F_2	Total
M_0	28.1	31.6	32.8	92.5	11.9	-9.2	13.0	15.7
M_1	33.2	37.4	29.6	100.2	11.6	19.8	3.4	34.8
M_2	29.8	31.9	29.5	91.2	16.8	-1.7	3.1	18.2
TOTAL	91.1	100.9	91.9	283.9	40.3	8.9	19.5	68.7

The values in the table are the sums and the differences, respectively, between the two halves of Table C-II.

CALCULATION OF VARIANCE

Correction term:

$$C = \frac{(\Sigma x)^2}{N} = \frac{(9.8 + \ldots\ 7.7)}{36} = \qquad 2235.7$$

Total sum of squares:

$$\Sigma X^2 - C = (9.8^2 \cdots 7.7^2) - 2235.7 = \qquad 862$$

Block sum of squares: 5 DF

$$\frac{\Sigma\ (\text{Block total})^2}{\text{no. in block}} - C = \frac{44.4 + \cdots 55.7}{6} - 2235.7 = \qquad 557$$

MAIN EFFECTS

Mixing sum of squares: 2 DF

$$\frac{\Sigma\ (\text{Mixing total})^2}{\text{no. results at each level}} - C = \frac{92.5^2 + \cdots 91.2^2}{12} - 2235.7 = \qquad 7$$

Flotation sum of squares: 2 DF

$$\frac{\Sigma\ (\text{Flotation total})^2}{\text{no. results at each level}} - C = \frac{91.1^2 + \cdots 91.9^2}{12} - 2235.7 = \qquad 8$$

Sea Water sum of squares: 1 DF

$$\frac{\Sigma\ (\text{sea water total})^2}{\text{no. results at each level}} - C = \frac{(\text{Difference between sea water levels})^2}{\text{no. results at both levels}} =$$

$$\frac{(68.7)^2}{36} = \qquad 131$$

(from bottom right corner of Table C-III)

INTERACTIONS

Mixing-Sea water sum of squares: 2 DF

a) Linear Effect

$$\text{Sum of squares} = \frac{(\Delta M_2 - \Delta M_0)^2}{12} = \frac{(18.2 - 15.7)^2}{12} = 0.5$$

(ΔM is the difference between the values of M (at a given level) at the two levels of sea water (with and without sea water). The ΔM values are found directly in the right hand column of Table C-III).

b) Curvature

$$\text{Sum of squares} = \frac{\left[2\Delta M_1 - (\Delta M_0 + \Delta M_2)\right]^2}{36} =$$

$$\frac{(2 \times 34.8 - 15.7 - 18.2)^2}{36} = 35.4$$

Average sum of squares = (0.5 + 35.4)/2 = 18

Flotation-Sea Water sum of squares: 2DF

a) Linear Effect

$$\text{Sum of squares} = \frac{(\Delta F_2 - \Delta F_2)^2}{12} =$$

$$\frac{(19.5 - 40.3)^2}{12} = 36$$

(The ΔF values are found in the bottom right hand row in Table C-III).

b) Curvature

$$\text{Sum of squares} = \frac{\left[2\Delta F_1 - (\Delta M_0 + \Delta M_2)\right]^2}{36} =$$

$$\frac{(2 \times 8.9 - 40.3 - 19.5)^2}{36} = 49$$

Average sum of squares = (36 + 49)/2 = 43

TABLE C-IV

ANALYSIS OF VARIANCE

Source of Variance	Degree Freedom	Sum of Squares	Mean Sum of Squares
Blocks & Replications	5	557	111
Mixing	2	7	8
Flotation	2	8	4
Sea Water	1	131	131**
Mixing - Sea water	2	18	9
Flotation - Sea water	2	43	22*
Mixing - Flotation, Third Order Interaction and Error	21'	98	4
Total	35	862	

**)Significant at 1% level. *)Significant at 5% level.

NOTES TO ANALYSIS OF VARIANCE:

The experiment was to consist of four replications, but based upon the analysis of the first two replications as shown above, it was decided instead to conduct the remainder of the experiment with another sewage. As it is shown in the analysis of variance the only significant effect is the sea water. With mixing and flotation being insignificant, their interaction cannot be expected to be significant, and could therefore not warrant the effort of completing the two remaining replications. The mixing-flotation interaction and the third order interaction cannot be calculated from two replications since both interactions were confounded over all four replicates.

B. SEWAGE WITH HIGH CONCENTRATION OF FLOATABLES

TABLE C-V

EXPERIMENT LAY-OUT AND RESULTS

Replicate I

Block a		Block b		Block c	
Code	mg/l	Code	mg/l	Code	mg/l
(200)	36.5	(000)	21.5	(100)	4.6
(010)	37.3	(110)	12.9	(210)	24.9
(120)	31.2	(220)	18.7	(020)	8.7
(101)	32.3	(201)	62.0	(001)	13.4
(211)	27.4	(011)	17.3	(111)	12.7
(021)	40.9	(121)	12.8	(221)	22.4

Replicate II

Block a		Block b		Block c	
Code	mg/l	Code	mg/l	Code	mg/l
(100)	19.1	(200)	16.1	(000)	25.4
(010)	19.0	(110)	24.6	(210)	29.3
(220)	15.6	(020)	16.7	(120)	24.1
(201)	13.9	(001)	17.5	(101)	25.1
(111)	8.3	(211)	14.1	(011)	13.1
(021)	12.9	(121)	13.6	(221)	19.3

TABLE C-VI

COMBINED TREATMENT TOTALS FOR REPLICATE I AND REPLICATE II

	S_0			S_1		
	F_0	F_1	F_2	F_0	F_1	F_2
M_0	21.5 25.4 46.9	37.3 19.0 56.3	8.7 16.7 25.4	13.4 17.5 30.9	17.3 13.1 30.4	40.9 12.9 53.8
M_1	4.6 19.1 23.7	12.9 24.6 37.5	31.2 24.1 55.3	32.3 25.1 57.4	12.7 8.3 21.0	12.8 13.6 26.4
M_2	36.5 16.1 52.6	24.9 29.3 54.2	18.7 15.6 34.3	62.0 13.9 75.9	27.4 14.1 41.5	22.4 19.3 41.7

Explanation: Top figure in each box is result from Replicate I, middle figure in each box is result from Replicate II, and bottom figure is the sum of the results from Replicates I and II. For example, the top figure in the box in the upper right corner is the amount of floatables from treatment code (021) in Replicate I and middle figure is result from code (021) in Replicate II.

TABLE C-VII

TWO-WAY TABLE

	$S_0 + S_1$				$S_0 - S_1$			
	F_0	F_1	F_2	Total	F_0	F_1	F_2	Total
M_0	77.8	86.7	79.2	243.7	16.0	25.9	-28.4	13.5
M_1	81.1	58.5	81.7	221.3	-33.7	16.5	28.9	11.7
M_2	128.5	95.7	76.0	300.2	-23.3	12.7	-7.4	-18.0
TOTAL	287.4	240.9	236.9	765.2	-41.0	55.1	-6.9	7.2

The values in the table are the sums and the differences, respectively, between the two halves of Table C-VI.

CALCULATION OF VARIANCE

Correction term:

$$C = \frac{(\Sigma x)^2}{N} = \frac{(36.5 + \ldots 19.3)^2}{36} = \qquad 16{,}265$$

Total sum of squares:

$$\Sigma x^2 - C = (36.5^2 + \ldots 19.3^2) - 16{,}265 = \qquad 4{,}033$$

Block sum of squares: 5 DF

$$\frac{\Sigma\,(\text{Block total})^2}{\text{no. in block}} - C = \frac{205.6^2 + \cdots 136.3^2}{6} - 16{,}265 = \qquad 1{,}712$$

MAIN EFFECTS

Mixing sum of squares: 2 DF

$$\frac{\Sigma\,(\text{Mixing total})^2}{\text{no. results at each level}} - C = \frac{243.7^2 + \ldots 300.2^2}{12} - 16{,}265 = \qquad 273$$

Flotation sum of squares: 2 DF

$$\frac{\Sigma\,(\text{Flotation total})^2}{\text{no. results at each level}} - C = \frac{287.4^2 + \cdots 236.9^2}{12} - 16{,}265 = \qquad 130$$

Sea water sum of squares: 1 DF

$$\frac{\Sigma\,(\text{Sea water total})^2}{\text{no. results at each level}} - C = \frac{(\text{Difference between sea water levels})}{\text{no. results at both levels}} =$$

$$\frac{(7.2)^2}{36} = \qquad 1.4$$

(from bottom right corner at Table C-VII)

INTERACTIONS

Mixing-Sea water sum of squares: 2DF

a) Linear effect

Sum of squares = $\frac{(\Delta M_2 - \Delta M_0)^2}{12}$ =

$\frac{(-18.0 - 13.5)^2}{12}$ = 82.8

b) Curvature

Sum of squares = $\frac{\left[2\Delta M_1 - (\Delta M_0 + \Delta M_2)\right]^2}{36}$ =

$\frac{(2 \times 11.7 - 13.5 + 18.0)^2}{36}$ = 21.6

Average sum of squares = (82.8 + 21.6)/2 = 52.2

Flotation-Sea water sum of squares: 2 DF

a) Linear effect

Sum of squares = $\frac{(\Delta F_2 - \Delta F_0)^2}{12}$ =

$\frac{(-6.9 + 41.0)^2}{12}$ = 96

b) Curvature

Sum of squares = $\frac{\left[2\Delta F_1 - (\Delta F_0 + \Delta F_2)\right]^2}{36}$ =

$\frac{(2 \times 55.1 + 41.0 + 6.9)^2}{36}$ = 694

Average sum of squares = (96 + 694)/2 = 395.

TABLE C-VIII

ANALYSIS OF VARIANCE

Source of Variance	Degree Freedom	Sum of Squares	Mean Sum of Squares
Blocks & Replications	5	1,712	342
Mixing	2	137	68
Flotation	2	65	33
Sea water	1	1	1
Mixing - Sea water	2	52	26
Flotation - Sea water	2	395	197
Mixing-Flotation, Third Order Interaction and Error	21'	1,671	80
Total	35	4,033	

NOTES TO CALCULATIONS

Like before, only the two first replicates were completed when the analysis showed that neither of the main factors was significant.

The very small sum of squares for the sea water main effect is mainly due to a single large value in the second block in the first replicate.

TABLE D-I

FLOATABLE MATERIAL IN INFLUENT AND EFFLUENT

Richmond Water Pollution Control Plant

First 24-Hour Period

Date & Time of Collection July 1965		INFLUENT Floatable Concentration mg/l	INFLUENT Flow Rate MGD	INFLUENT Weight of Floatables lbs.	EFFLUENT Floatable Concentration mg/l	EFFLUENT Flow Rate MGD	EFFLUENT Weight of Floatables lbs.
13	0900	3.3	9.5	21.8[1]	3.7	6.0	15.4
13	1100	0.2	9.4	1.3	1.8	9.5	11.9
13	1300	4.8	9.2	30.7	3.3	9.4	21.6
13	1500	6.2	7.0	30.2	2.7	9.2	17.3
13	1700	12.1	6.1	51.3	2.5	7.0	12.2
13	1900	4.9	6.5	22.2	3.7	6.1	14.4
13	2100	2.7	8.1	15.2	3.2	6.5	14.5
13	2300	5.3	7.0	25.8	2.1	8.1	11.8
14	0100	2.4	5.0	8.3	0.6	7.0	2.9
14	0300	4.4	4.5	13.8	2.0	5.0	7.0
14	0500	8.6	3.8	22.7	1.4	4.5	4.4
14	0700	7.1	5.5	27.1	2.2	3.8	5.8
Total Amount of Floatable Material				270.4 lbs			134.5 lbs.

1) Pounds of floatable material during 2-hour period = mg/l x MGD x $\frac{2}{24}$ x 8.34

TABLE D-I Cont'd.

FLOATABLE MATERIAL IN INFLUENT AND EFFLUENT

Richmond Water Pollution Control Plant

Second 24-Hour Period

	INFLUENT			EFFLUENT		
Date & Time of Collection July 1965	Floatable Concentration mg/1	Flow Rate MGD	Weight of Floatables lbs.	Floatable Concentration mg/1	Flow Rate MGD	Weight of Floatables lbs.
16 0900	11.9	9.4	77.7	5.5	5.8	22.2
16 1100	4.6	9.0	28.8	4.2	9.4	27.4
16 1300	3.8	10.0	26.4	2.7	9.0	16.9
16 1500	3.3	8.5	19.5	2.2	10.0	15.3
16 1700	4.3	8.0	23.9	2.3	8.5	13.6
16 1900	9.4	7.8	1.0	1.5	8.0	8.3
16 2100	1.4	7.6	7.4	0.8	7.8	4.3
16 2300	3.9	5.8	18.5	1.9	7.6	10.0
17 0100	4.8	5.0	16.7	0.9	5.8	3.6
17 0300	4.9	2.8	9.5	1.7	5.0	5.9
17 0500	3.8	2.2	5.8	1.3	2.8	2.5
17 0700	2.4	5.8	9.7	1.3	2.2	2.0
Total Amount of Floatable Material			294.9			132.0

TABLE D-II

FLOATABLE MATERIAL IN RAW SLUDGE

Richmond Water Pollution Control Plant Survey

FIRST 24-HOUR PERIOD				SECOND 24-HOUR PERIOD			
Date & Time of Collection July 1965	Floatable Concentration mg/l	Flow Rate gal/hr	Weight of Floatables lbs.	Date & Time of Collection July 1965	Floatable Concentration mg/l	Flow Rate gal/hr	Weight of Floatables lbs.
13 0900	40.0	430	0.3	16 0900	272.0	1,000	4.5
13 1100	65.0	600	0.7	16 1100	169.0	1,000	2.8
13 1300	76.5	750	1.0	16 1300	101.0	1,000	1.7
13 1500	99.5	750	1.2	16 1500	72.0	1,000	1.2
13 1700	57.0	750	0.7	16 1700	52.5	1,155	1.0
13 1900	67.5	750	0.8	16 1900	53.5	1,000	0.9
13 2100	440.0	750	5.5	16 2100	58.5	750	0.7
13 2300	354.5	375	2.2	16 2300	311.5	430	2.2
14 0100	109.0	375	0.7	17 0100	268.0	430	1.9
14 0300	180.5	375	1.1	17 0300	296.0	430	2.1
14 0500	100.5	375	0.6	17 0500	171.0	430	1.3
14 0700	101.5	375	0.7	17 0700	149.0	430	1.1
Total Amount of Floatable Material			15.5	Total Amount of Floatable Material			21.4

TABLE D-III

MOISTURE CONTENT OF FLOATABLE MATERIAL COLLECTED AT RICHMOND WATER POLLUTION CONTROL PLANT JULY 1965

Date and Time	Initial Net Weight of Sample (kg)	Net Weight of Sample After Drying at 40°C (kg)	Dry Material at 40°C Percent	Percent HEM*) of Dry Matter
13 1030	1.52	0.70	46%	
13 1740	0.76	0.35	46%	
14 0100	1.92	0.80	42%	
14 0700	0.72	0.51	71%	
Average of first 24-hour period			51%	98%
Composite for period 160900 - 170700	1.21	0.57	47%	93%

*)Hexane Extractable Material determined on dry sample by direct Soxhlet extraction.

TABLE E-I

FLOATABLE MATERIAL SURVEY

South San Francisco-San Bruno Water Pollution Control Plant

First 24-Hour Period

Date & Time of Collection Aug-Sept 1965	INFLUENT Floatables Concentration mg/1	INFLUENT Flow Rate MGD	INFLUENT Weight of*) Floatables lbs.	EFFLUENT Floatables Concentration mg/1	EFFLUENT Flow Rate MGD	EFFLUENT Weight of Floatables lbs.
30 0900	8.5	8.8	52.0	0.8	8.8	4.9
30 1100	7.1	12.5	61.7	1.5	12.5	13.0
30 1300	7.7	11.8	63.1	0.5	11.8	4.1
30 1500	3.2	10.7	23.8	0.9	10.7	6.7
30 1700	3.1	9.5	20.5	0.1	9.5	0.7
30 1900	4.4	9.5	29.1	0.8	9.5	5.3
30 2100	3.2	9.5	21.1	2.5	9.5	16.5
30 2300	2.7	7.6	14.3	1.0	7.6	5.3
31 0100	4.9	5.0	17.0	0.2	5.0	0.7
31 0300	5.0	3.2	11.1	0.1	3.2	0.2
31 0500	19.9	4.8	66.4	0.3	4.8	1.0
31 0700	3.7	4.5	11.6	0.3	4.5	0.9
Total Amount of Floatables			391.7			59.3

*)Pounds of floatable material during 2-hour period = mg/1 x MGD x $\frac{2}{24}$ x 8.34

TABLE E-I Cont'd

Second 24-Hour Period

Date & Time of Collection Aug-Sept 1965		INFLUENT Floatables Concentration mg/1	INFLUENT Flow Rate MGD	INFLUENT Weight of Floatables lbs.	EFFLUENT Floatables Concentration mg/1	EFFLUENT Flow Rate MGD	EFFLUENT Weight of Floatables lbs.
31	0900	3.5	8.5	20.7	0.4	8.5	2.4
31	1100	6.1	10.5	44.5	0.7	10.5	5.1
31	1300	4.3	10.5	31.4	0.7	10.5	5.1
31	1500	6.3	10.3	45.1	0.6	10.3	4.3
31	1700	3.4	9.5	22.4	0.1	9.5	0.7
31	1900	6.7	9.0	41.9	0.8	9.0	5.0
31	2100	3.5	9.0	21.9	0.5	9.0	3.1
31	2300	4.6	7.2	23.0	0.8	7.2	4.0
01	0100	4.0	6.0	16.7	(-0.1)	6.0	0.0
01	0300	3.6	3.1	7.8	0.4	3.1	0.9
01	0500	2.8	3.0	5.8	0.4	3.0	0.8
01	0700	1.9	4.0	5.3	0.4	4.0	1.1
Total Amount of Floatables				286.5			32.5

TABLE E-I Cont'd.

Third 24-Hour Period

Date & Time of Collection Sept. 1965		INFLUENT			EFFLUENT		
		Floatables Concentration mg/1	Flow Rate MGD	Weight of Floatables lbs.	Floatables Concentration mg/1	Flow Rate MGD	Weight of Floatables lbs.
02	0900	3.2	10.0	22.2	0.2	10.0	1.4
02	1100	105.3+	10.5	215.6*	0.4	10.5	2.9
02	1300	5.3	10.3	37.9	0.5	10.3	3.6
02	1500	6.9	9.3	44.6	1.4	9.3	9.0
02	1700	5.7	9.0	35.7	0.0	9.0	0.0
02	1900	9.6	10.8	72.1	0.7	10.8	5.3
02	2100	12.2	8.5	72.1	0.7	8.5	4.1
02	2300	3.4	7.9	18.7	0.1	7.9	0.6
03	0100	8.1	5.0	28.1	0.2	5.0	0.7
03	0300	3.1	3.3	7.1	0.2	3.3	0.5
03	0500	5.2	2.6	9.4	0.2	2.6	0.4
03	0700	4.9	3.6	12.3	0.1	3.6	0.3
Total Amount of Floatables				575.8			28.8

+) Visual inspection of the influent stream showed that this high concentration occurred during a period of approximately 30 minutes and 1/4 of value used in calculation of average concentration.

*) Total weight calculated from 105.3 mg/1 for 30 minutes and an estimated 4.3 mg/1 for the remainder 180 minutes of the two-hour period.

TABLE E-II

FLOATABLE MATERIAL IN SKIMMINGS AT SSF-SB WATER POLLUTION CONTROL PLANT

Date & Time of Collection Aug-Sept 1965		Floatables Concentration mg/l	Estimated Flow Rate MGD	Total Amount of Floatable Material
30	0900	7,596	0.02	
30	1300	2,636	0.02	
30	1700	1,936	0.02	
30	2100	2,710	0.02	
31	0100	1,338		
31	0500	550	0.02	
Average		2,800	0.02	467 lbs.
31	0900	754	0.02	
31	1300	4,658	0.02	
31	1700	2,168	0.02	
31	2100	1,970	0.02	
01	0100	1,580	0.02	
01	0500	3,790	0.02	
Average		2,490	0.02	415 lbs.
02	0900	2,952	0.02	
02	1300	11,303	0.02	
02	1700	9,595	0.02	
02	2100	4,180	0.02	
03	0100	3,423	0.02	
03	0500	3,120	0.02	
Average		5,760	0.02	961 lbs.